

THESIS FOR THE DEGREE OF LICENTIATE OF ENGINEERING

# **Analysis and Modelling of Mineral and Element Composition in Compression Breakage**

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Cover:

The cover illustration is a backscattered electron image of a composite particle after a compressive breakage process. The particle is embedded in by tantalite, quartz, muscovite and albite.

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To my family



# ABSTRACT

The outstanding properties of tungsten and tantalum make them valuable metals and, in some cases, irreplaceable in applications. The growing interest due to the high economic importance and the limited supply places these metals in the critical metals risk list. As a consequence of the increasing global demand for these metals, there is also a need to develop more efficient extraction processes. Coarse comminution processes are commonly assessed by the size reduction of particles, however, liberation and mineralogy are not taken into consideration.

The main hypothesis of this research is that critical metals are not evenly distributed in the different size fractions during coarse comminution processes since the breakage of particles will be affected by the mineralogy and texture. Further, to demonstrate the significant impact that mineralogy understanding has during breakage and develop a test procedure to include the concentration of the critical metals which will increase the resolution of coarse comminution models.

This thesis focuses on finding an experimental methodology to analyse the rock properties and its general characteristics, followed by defining a multicomponent model that combines size reduction and concentration for critical metals. In this research, the work is divided into 3 stages. The first main stage is the rock materials characterisation which consists of two parts: mechanical and mineralogical analyses. The second stage is the modelling and the third stage is theoretic implementation.

The mechanical characterisation includes compressive breakage through the use of interparticle breakage. Mineralogical characterisation was evaluated through the assessment of size fractions with a scanning electron microscopy (SEM) and geochemical analysis. Results from these tests give information about breakage, mineral composition and element concentration.

The second main stage corresponds to modelling. The first modelling part was the methodology of fitting measured data into a size reduction model. The second part corresponds developing a model capable of predicting the rare metal concentration as a function of the particle size distribution after a cycle of the compression crushing process. The model was developed by selecting a bimodal Weibull distribution for calibration which is shown to be capable of simulating critical metal concentration as a function of the compression ratio.

The third stage is a simulation. The aim of this section is to demonstrate a theoretical case where the concentration model allows to make well based estimations of how a plant should be designed. Evaluations have shown that by considering the information of the ore and its behaviour in terms of concentration and performance of the crushing, the process could be improved by changing machine parameters, i.e. closed side setting and adding a pre-screening before the milling process.

The work shows that considering the effect of mineralogy and element concentration in the coarse comminution models, it is possible to achieve better performance in terms of cone crusher and plant design.

**Keywords:** compressive crushing, modelling, rare metals, cone crusher, minerals, elements.



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Por último, sin embargo el más importante, gracias a Dios por hacer todo posible en mi vida.

Lorena Guldri Leon  
Gothenburg, Sweden, April 2018





# APPENDED PUBLICATIONS

The licentiate thesis contains the following papers:

- Paper A: Guldris, L., Bengtsson, M., and Evertsson, C. M. *Reduction and fracture analysis of a tungsten ore and its use for fundamental liberation modelling*. 12<sup>a</sup> Conferencia Internacional de Procesamiento de Minerales (PROCEMIN 2016). 2016. Santiago, Chile.
- Paper B: Guldris, L., Bengtsson, M., and Evertsson, C. M. *Mineralogical characterisation, reduction and liberation analysis of tungsten ore*. Emerging Trends in Minerals Engineering Conference (IOM<sup>3</sup>). 2016. London, U.K
- Paper C: Guldris, L., Bengtsson, M., and Evertsson, C. M., *Modelling Reduction and Liberation for Rare Earth Minerals Applications*. International Comminution Symposium (Comminution '16). 2016. Cape Town, South Africa.
- Paper D: Guldris, L., Bengtsson, M., and Evertsson, C. M. *Analysis of concentration in rare metal ores during compression crushing*. International Journal of Mineral Engineering (Journal). January 2018.

*In papers A-C, Guldris and Bengtsson initiated the idea. The testing was performed by Guldris. Guldris wrote the paper with Evertsson as reviewer.*

*In papers D, Guldris and Bengtsson initiated the idea and co-wrote the paper with Evertsson as reviewer.*



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## APPENDED PAPERS

Paper A: Reduction and fracture analysis of a tungsten ore and its use for fundamental liberation modelling.

Paper B: Mineralogical characterisation, reduction and liberation analysis of tungsten ore.

Paper C: Modelling Reduction and Liberation for Rare Earth Minerals Applications.

Paper D: Analysis of concentration in rare metal ores during compression crushing.

# LIST OF ABBREVIATIONS

CRPS	Chalmers Rock Processing Systems	
CSS	Close Side setting	
BCA	Bulk chemical analysis	
EDS	Energy Dispersive Spectroscopy	
ICP-AES	Inductively Coupled Plasma Mass Spectrometry	
ICP-MS	Inductively-Coupled Mass Spectrometry	
IPB	Interparticle Breakage	
SEM	Scanning Electron Microscopy	
SPB	Single particle Breakage	
QMA	Quantitative Microstructural Analysis	
VSI	Vertical Shaft Impactor	
$b$	Bed height	[mm]
$s$	displacement of the piston	[mm]
$s/b$	Compression ratio	[-]
$x_{s/b}$	Normalized particle size	[-]
$x$	Particle size	[mm]
$B$	Breakage matrix	[-]
$f$	Feed distribution	[mm]
$I$	Identity matrix	[-]
$p$	Product distribution	[mm]
$S$	Selection matrix	[-]
$\alpha$	Calibration constant	[-]
$\beta$	Calibration constant	[-]
$\gamma_o$	Normalized frequency distribution concentration	[-]
$\nu$	Calibration constant	[-]
$\lambda$	Calibration constant	[-]
$d$	Integration interval fraction size	[mm]
$c$	Integration interval fraction size	[mm]
$i$	Element index	[-]
$m_i$	Mass in each fraction	[g]
$m_i \text{ ot}$	Total mass	[g]
$p_i$	Size fraction	[-]
$H_0$	Cumulative normalized distribution concentration	[-]





# INTRODUCTION

*The aim of this chapter is to:*

- *Introduce minerals and mineral processing.*
- *Provide an overview of comminution processes.*
- *Introduce the principles of breakage.*
- *Provide an overview of cone crushers.*
- *Define liberation of valuable minerals.*
- *Describe challenges associated with coarse comminution processes.*

Tungsten and tantalum are valuable and irreplaceable metals with multiple ranges of applications in modern technologies ranging from industrial to electronic applications. This is due to the properties that these metals have, such as the high density, the highest melting point of all metals, low reactivity, and also when combined with other material they are well known for improving mechanical properties as hardness, toughness and corrosion resistance. Tungsten and tantalum are raw materials with very high economic importance and with a medium supply risk, which is the reason why the extraction of these metals is positioned in the top of the “risk list”, from the British Geological Survey (2012) immediately after the rare earth elements.

These critical metals, like all metals, are extracted from ores through processes that include numerous steps, beginning with size reduction of the ore particles through to separation. The size reduction of the particles is known as comminution, and it includes crushing (coarse comminution) and grinding (fine comminution).

## 1.1 MINERALS AND MINERAL PROCESSING

Minerals are everything with an economic value which is extracted from the earth. The minerals are found in ores or rocks which are typically not homogeneous in chemical and physical composition. Normally, minerals are finely disseminated and intimately associated with gangue which has no commercial value. To achieve the extraction of the valuable minerals it is necessary to process the ore through different steps including size reduction and physical separation. Mineral processing refers to the different necessary processes for the extraction of valuable metals and produces a commercial end product or products.

There are two fundamental operations in mineral processing, the liberation or release of the valuable minerals from their waste gangue minerals, and the separation into two products one the concentrate that is the enriched-product with most of the valuable minerals, and the tailings which are the discard product that contains predominately the gangue minerals. Figure 1 illustrates the simple flowsheet of mineral processing process.



Figure 1. Principle flowsheet of the mineral processing stages.

The liberation of the valuable minerals is achieved by the reduction of particles in sizes through comminution processes. It is necessary to reduce the size particles to reach a mixture of relatively clean and pure particles of either mineral or gangue (Wills and Finch, 2016). Figure 2 illustrates a more detailed flowsheet of the mineral processing process focusing on the interactions during the comminution stage.



Figure 2. Flowsheet of the mineral processing process.

The comminution circuit is comprises crushing, grinding and initial rejection. Usually, a comminution plant consists of two to three crushing stages and one to three milling stages, as Figure 3 illustrates. The purpose of each stage of is to reduce the particle size to a certain degree. When the particles reach the desired reduction, they are transferred to the next process to be reduced to a smaller size until achieving the required size. Usually, the primary crusher is a jaw or gyratory crusher, secondary crushers can be of gyratory or cone type, while tertiary crushers are cone crushers (Bengtsson, 2009). Normally the design of a plant depends on several factors including ore grade, ore type, ore deposit variability, the scale of the operation and investment capital.



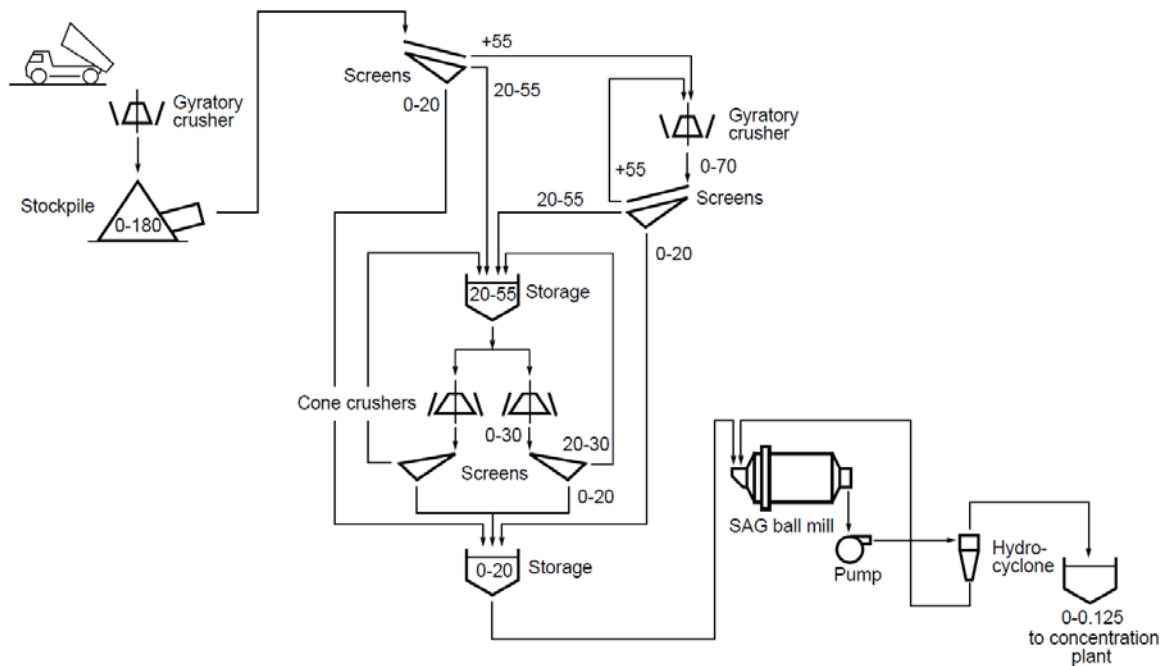


Figure 3. Size reduction circuit for a nickel rich ore (Evertsson, 2000).

## 1.2 COMMUNITION

Comminution is the process where the ore particles are reduced in size. The size reduction is carried out to liberate or release the valuable minerals from gangue so that the separation process can take place in the following step (Wills and Finch, 2016). The comminution process involves a high amount of energy since it requires that all the particles are reduced in size. Approximately, 2% of all electrical energy generated worldwide is used in comminution process (Napier-Munn, 2015).

Comminution in mineral processing plants takes place as a sequence of crushing (coarse comminution) and milling processes (fine comminution). The crushing stages reduce the particle size from run-of-mine ore to such a level that grinding can be carried out, until the valuable mineral and gangue particles can be separated from each other. Comminution of a rock material can be achieved by three different breakage mechanisms: compression, impact and attrition. Normally during the crushing stage, the reduction of the particles is accomplished by compression or impact, while during milling the used breakage mechanism is abrasion and impact. Generally, crushing is carried out as a dry process, while milling is usually performed in wet conditions (Wills and Finch, 2016).

Milling requires and consumes the highest amount of energy that could represent up to 50% of a concentrators total energy consumption. The problems associated with fine grinding are not only the energy cost, but also this process can lead to very fine size particles, which are untreatable and may be lost into the tailings (over-grinding). In low-grade ores, where it is necessary to achieve very small sizes, the energy cost for fine comminution and the fines losses can be significantly high (Wills and Finch, 2016).

The evaluation of the quality and performance of coarse comminution processes are normally assessed by measuring the particle size distribution. Measuring of the mineral liberation is just considered at the end of the fine comminution process.

### 1.3 BREAKAGE PRINCIPLES

There are three main comminution principles of industrial interest:

- Impact breakage
- Attrition
- Compressive breakage

The three principles are shown in Figure 4. In the sub-sections below the different mechanisms will be described.

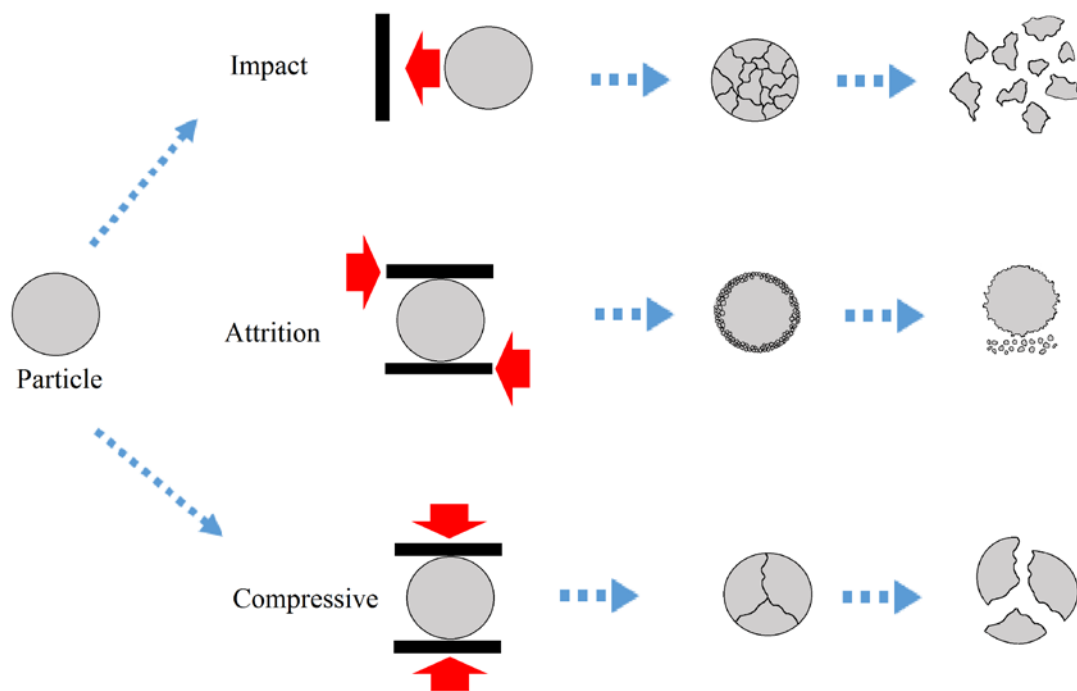


Figure 4. Particle breakage mechanisms, forces present in the particles and the resultant distribution of broken particles

#### 1.3.1 Impact breakage

During impact breakage, the energy is transmitted to the particles as kinetic energy by the acceleration. The fragmentation occurs when the particles impact with walls or other particles. Figure 4 shows the basic principle of this type of breakage, as well as the resultant particle size distribution. The size reduction is controlled by the amount of energy transmitted to the particle which, at the same time, is dependent on the speed of the rotor. For this reason the breakage could be controlled by the speed. Crusher equipment using this principle are known as impact crushers. Some equipment that work with this principle are: the vertical shaft impactor (VSI), hammer mill and impact mill. The purpose of this type of crushers is to reduce the size particle,

but during the process, the generation of fine particles is high due to the abrasive forces present which is sometimes required but sometimes undesirable (Evertsson, 2000).

### 1.3.2 Attrition

Attrition forces are caused by shear failure and occur due to the interaction between particles. The attrition mostly generates fine material. Figure 4 shows the basic principle of attrition in breakage, as well as the resultant particle size distribution. The attrition is a result of impact, compression or shear forces, or the combination of them.

### 1.3.3 Compressive breakage

During compressive breakage, the rock material is crushed by compression forces between two surfaces. In this type of breakage, the contact forces between the particles create an increase in the tensile stresses in the particles. When the tensile stresses exceed a critical value, cracks are generated that propagate and break the rock into smaller pieces. The compression forces are achieved through the displacement of the surfaces (or one surface). It is possible to regulate the size reduction by controlling the compression, using the forces and input energy. Some examples of crushing equipment that uses compressive breakage as a principle to achieve particle size reduction are jaw, gyratory, cone and roller crushers. The compressive forces could be applied one at a time or in a repetitive process. As a consequence of the compressive forces the particles will break, the shape and the size of these broken particles can be divided into two size classes. Some larger fragments formed by the fractures and cracking as a consequence of the tensile stress state will be generated, but also some amount of fine particles will be generated through the shear stresses in the contact points. The principle of compressive breakage and forces present are illustrated in Figure 5 (Evertsson, 2000).

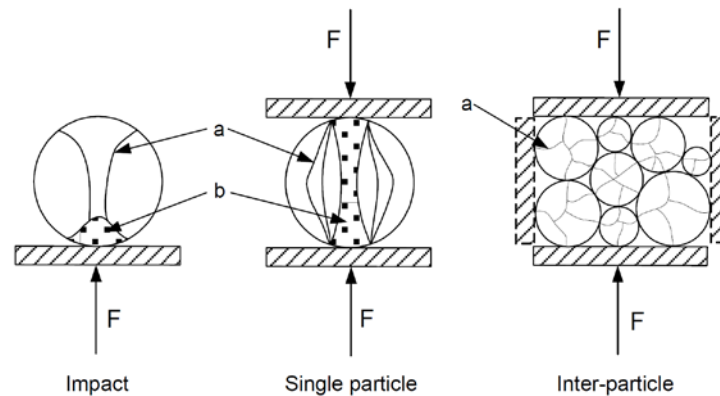


Figure 5. Breakage principles. a) Description of cracks and fractures generated by tensile stress. b) Description of fine particles generated through shear stress. (Bengtsson, 2009)

## 1.4 CONE CRUSHERS

For secondary and tertiary crushing stages, the most commonly used equipment is the so called cone crusher. Cone crushers use the compressive breakage principle as a functional principle. During the reduction process in this machine, the particles are subjected to single particle breakage (SPB) and interparticle breakage (IPB) throughout the different steps of the process. The compression of the particles is achieved by the movement of the inner cone (mantle) while the outer cone (concave) is fixed.

It is possible to control the final particle size by controlling the distance between the mantle and the concave. This machine parameter is known as closed side setting (CSS). There are different types of cone crushers on the market, all of them use the same principle but with different design or sizes. The choice of crusher size and chamber design depends on the requirements regarding of capacity, material and feed particle distribution. Figure 6 shows a general cone crusher and its design.

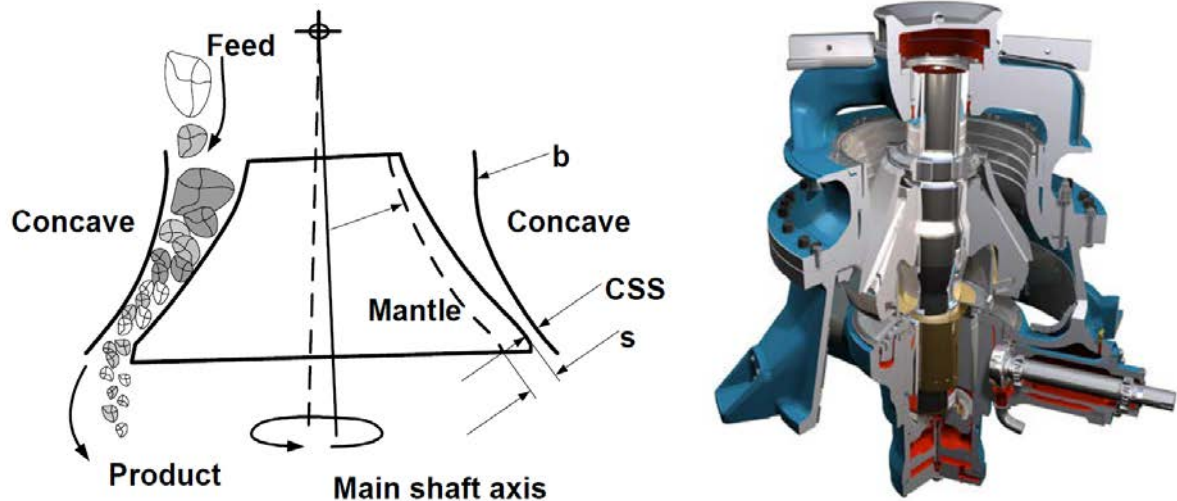


Figure 6. Cone Crusher (Bengtsson, 2009).

## 1.5 LIBERATION OF VALUE MINERALS

As previously explained, the objective of comminution is to liberate or release the valuable minerals from the gangue minerals as coarsely as possible to facilitate the separation in following processes.

The liberation is measured by the percentage of the mineral occurring as free particles in the ore in relation to the total content. This concept is known as the degree of liberation. Complete liberation between mineral and gangue particles is seldom achieved, even when the ore is reduced to the desired grain size. The properties of the grains and the grain boundaries determine the difficulties to achieve the complete liberation.

Depending on the process and separation techniques, a high degree of liberation is not necessary for achieving high recovery values. In general, a gravity separation process does not need particles with a high degree of liberation, if a pronounced density is apparent between the locked particles and the free gangue particle, the separation is possible. The same happens during magnetic separation processes, due to the magnetic susceptibility difference between gangue and valuable mineral (Wills and Finch, 2016).

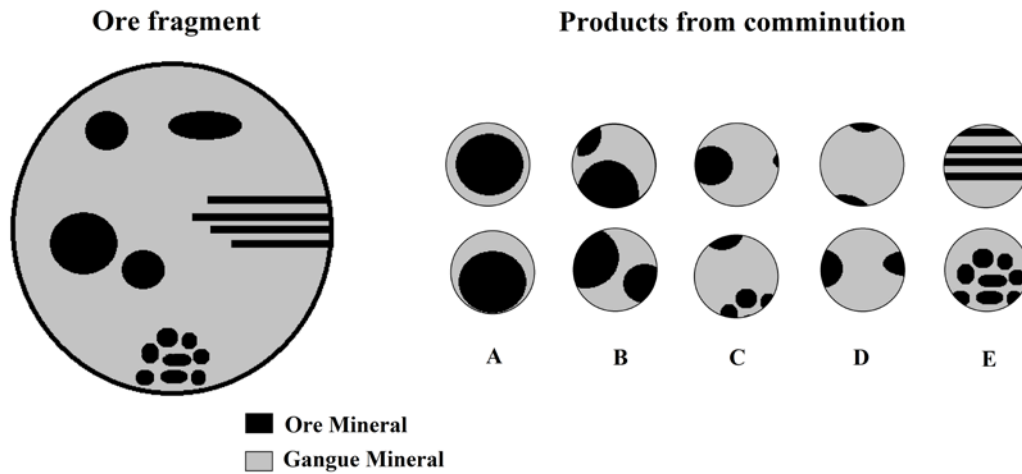


Figure 7. Ore particles. (Wills and Finch, 2016)

Figure 7 shows an ore particle, it is possible to identify two section in the particle. One rich in the valuable mineral (black section), and other rich in the valuable mineral but highly interspersed with the gangue mineral. The reduction of this particle through comminution process will generate liberated fragments of mineral and gangue particles, but will also generate composite particles with different degrees of valuable minerals. Depending on the degree or amount of valuable mineral, the particles will be classified as a concentrate (In Figure 8, particles similar at A), tailings (particles D) or middlings (particles B, C and D) (Wills and Finch, 2016).

During liberation analysis, the size reduction of the particles is not the only interesting concept. In this stage it is also important to understand how the minerals break. The responsibility of the mineral liberation was first attributed by A. M. Gaudin (1939), who described two types of mineral liberation: by comminution and by detachment. Liberation by comminution or random breakage, is the liberation achieved by random fracture mechanism, meaning the crack propagation is through the mineral grains without consideration of the different phases present in the ore. In this type of breakage, the particles from a single size class will be broken into a predictable unimodal distribution of finer particles, allowing the composition of all particles to remain constant. In this case, the ore texture, grain boundaries or mineral properties do not influence the breakage.

The second type is liberation by detachment or non-random breakage. In this type of breakage the fractures or cracks propagation are situated along the grain boundaries allowing the preferential liberation of the phases, also known as an intergranular fracture. This will generate a different concentration in the composition of different size fractions allowing the uses selective comminution techniques. There are different definitions of non-random breakage, however, regardless of their classification, they show a variation in the mineral composition of different size fraction.

There are two forms of non-random breakage in particle bed breakage: interfacial breakage (grain/phase boundary fracture) and preferential breakage. In interfacial breakage, the fracture occurs between different grains or between the phase boundaries, allowing the preferential liberation of the phases. In the second case, preferential breakage occurs when the characteristics of the phases are different, resulting in breakage at different rates according to the soft mineral and hard mineral contents of the ore. The soft minerals will break more rapidly

than the hard minerals, considering the same amount of forces, generating different concentrations in the composition of the fine fraction and the coarse fraction (Fandrich, 1997); (Mariano, 2016); (Hesse, 2017).

## 1.6 CHALLENGES ASSOCIATED WITH COARSE COMMINUTION PROCESS

Comminution processes involve a high amount of energy to assure the optimal size range of the ore particles until the separation. The evaluation of the quality and performance of coarse comminution processes are normally assessed by measuring the particle size distribution. Measuring the mineral liberation and composition is just considered at the end of the process.

All of the minerals that form the ore exhibit diverse characteristics and properties, including grain size, grain shape, bounding between grains and mechanical properties of the minerals such as Young's modulus and strength. All of these properties may affect how the ore is broken and the result achieved after the breakage process, by means the particle size distribution Vizcarra et al. (2010). Recently, the early rejection of the coarse materials has been identified as an alternative to upgrading the process, especially to decrease energy consumption. The early rejection or preferential grade by size, is based on the propensity of some ores to deport metal into specific size fractions (Carrasco, 2016a; Carrasco, 2016b), but to achieve this it is necessary to take advantage of the variances between the minerals phases that integrate the ore. To recognize these differences, it is important to identify some concepts such as grain shape, grain size, crack propagation, breakage mechanism and element concentration (Hesse, 2017).

Significant advances in comminution technologies may come from the exploitation of the basic fundamental understanding of the fracture process. Demonstrating the significant impact of phase boundary, breakage mechanism and element concentration increase the resolution of the process.

# 2

## OBJECTIVES

*The goal of this chapter is to:*

- *Describe the aim of this research.*
- *Formulate the research questions.*
- *Define the delimitations of the research.*

### 2.1 RESEARCH OUTLINE

The aim of this work is to gain fundamental understanding of how different ore materials break after compressive crushing in terms of minerals and elements distribution. Consequently, the main hypothesis of this research is that critical metals are not evenly distributed in different size fractions during coarse comminution processes since the breakage of particles will be affected by the mineralogy and texture.

Further, the aim of this work is to understand how ore characteristics can be measured and integrated into coarse comminution models. The specific objective of this work is to develop a test procedure capable of predicting the critical metal concentration as a function of the particle size distribution after a cycle of the compression crushing process.

The material characterisation process and methodology for the analysis is shown in Figure 8. The research focuses on compressive breakage in tertiary crushing stages, as shown in Figure 9.

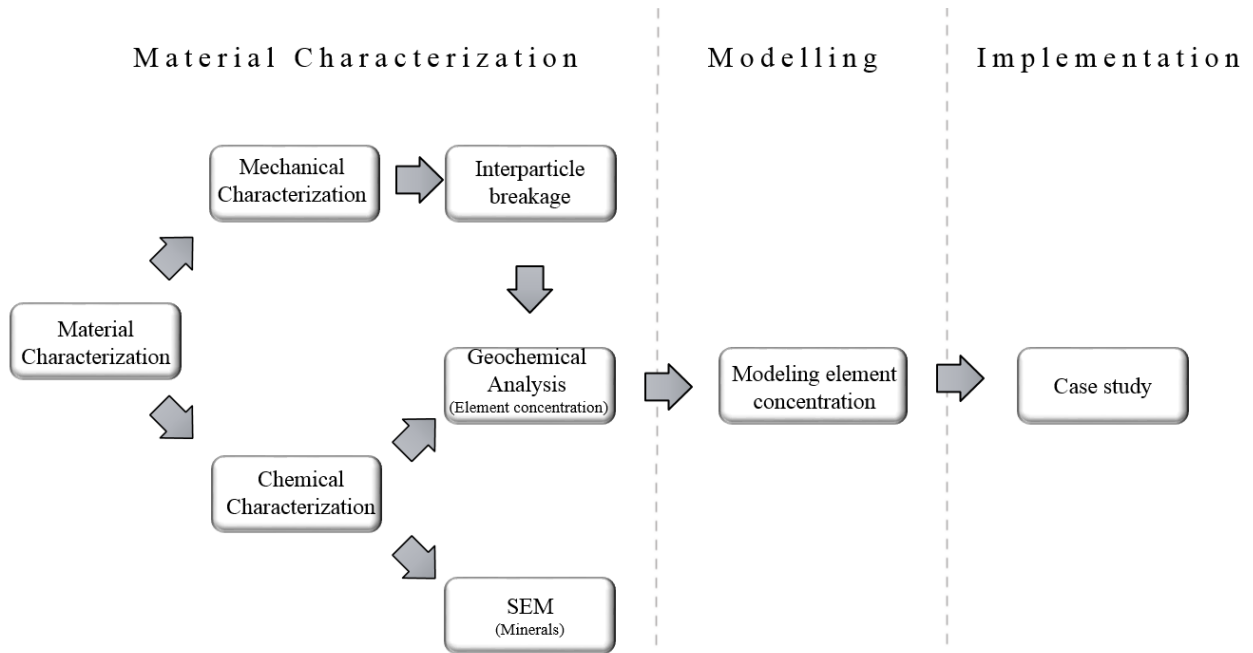


Figure 8. Material characterisation test procedure.

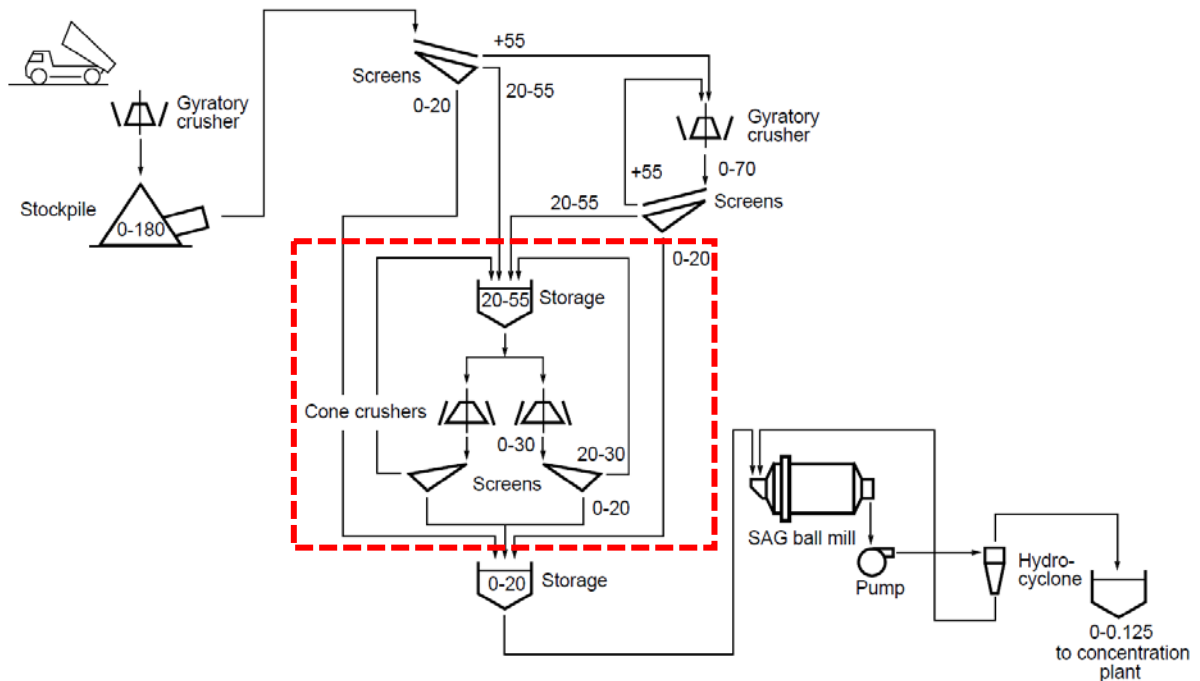


Figure 9. The focus of this work is the tertiary crushing in mineral plants as shown in the dashed box (Evertsson, 2000).





















## 2.2 RESEARCH QUESTIONS

A set of research questions has been formulated to guide the research work. The focus of the research questions varies from material characterisation to modelling.

- RQ 1)* What type of material properties beside particle size need to be analysed to model element grade?
- RQ 2)* How can liberation in coarse comminution be measured?
- RQ 3)* Is it possible to achieve liberation of critical metals in coarse comminution?
- RQ 4)* What factors influence the concentration distribution and how can it be measured?
- RQ 5)* How can concentration of critical metals be modelled?
- RQ 6)* How can coarse comminution improve the extraction of critical metals?

The research questions are going to be discussed throughout this thesis and will be answered in the conclusions chapter. The relation and dependency of the research questions and the appended papers are shown in Figure 10. The figure refers to the degree to which the research questions are addressed in different chapters.

	RQ1	RQ2	RQ3	RQ4	RQ5	RQ6
Paper A						
Paper B						
Paper C						
Paper D						



 Strong contribution     
  Medium contribution

Figure 10. Relationship between research questions and papers.

## 2.3 DELIMITATIONS

In order to limit the scope of the research a set of delimitations have been formulated as listed below:

- The work is based on compressive breakage.
- The work is based on the study and characterisation of critical metal ores. The modelling of the concentration was validated for tantalum and tungsten ores.
- The work does not include screening and classification
- This work is not including on dynamic process simulations.



# 3

## SCIENTIFIC APPROACH

*The aim of this chapter is to:*

- *Introduce the methodology used in the research.*

This work has been carried out at the Chalmers Rock Processing Systems (CRPS) research group at the Department of Industrial and Materials Science at Chalmers University of Technology. CRPS is a part of the Machine Elements group. The research topic addressed by the group is focused on processes for producing crushed rock materials applied to the mining and aggregate industry.

The adopted scientific approach used by the Machine Elements group is characterized as a problem-oriented research methodology. In problem-oriented research approaches the methods selected for solving the problem are based on the nature of the problem rather than tools or methods required to solve it. The method has been thoroughly described by Evertsson (2000) and Hulthén (2010). The schematic diagram of the problem-oriented research methodology is shown in Figure 11.

Research is defined as a detailed study to discover new information or reach a new level of understanding. The need for the new understanding or new knowledge is based on a problem that requires a solution-finding where the existing knowledge is not sufficient and a deeper understanding is needed. The first step in the process is to identify the lack of knowledge. The next step observes the reality and the physical principles that are related to the specific problem. Different kinds of observations are used for this purpose. The literature reviews are used as the basis of the existing knowledge in the research area and experiments can be used to clarify the nature of the problem. The data acquisition or experiments can be evaluated and used in the process of building theory.

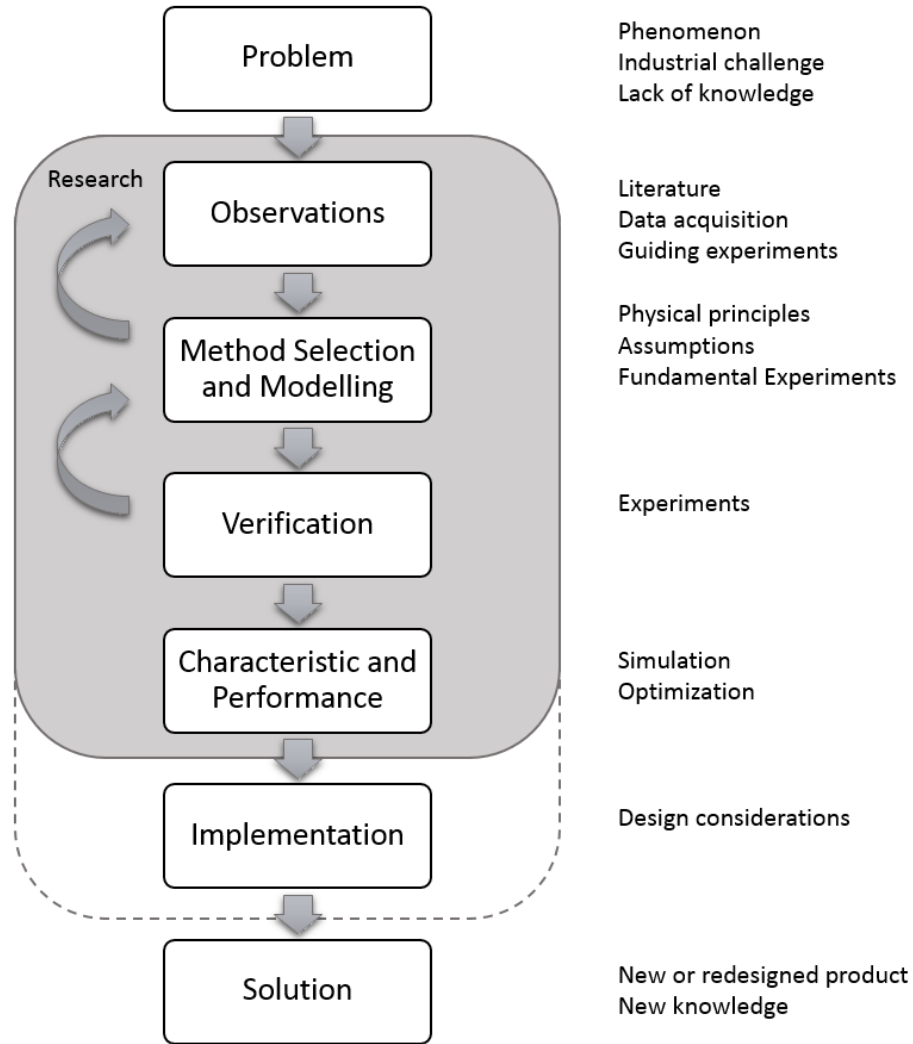


Figure 11. Structure of problem-based research approach

Consequently, methods and models are evaluated and developed. The models are used to describe the phenomena in mathematical formulations of physical measurement of behaviour that has been discovered. The idea is to select the most suitable method and model according to the nature of the problem it is therefore necessary to use an iterative process where several potential methods are tested in order to find the best one. The result of this stage is then tested and verified, preferably together with the industry or by using a case study. Even this step is an iterative process where new interactions could lead to potential improvements. With the help of the verified models, simulations can be performed to get more information, knowledge and improvement of the performance. The simulation will contribute to new conclusions which consider important effects and how the process could be improved. The new knowledge is created and can be implemented. The result of this process is the solution of the original problem.

# 4

## LITERATURE REVIEW

*The aim of this chapter is to:*

- *Explain the traditional compressive breakage models.*
- *Describe the bases of liberation models.*
- *Describe fundamental concepts.*

### 4.1 COMPRESSIVE BREAKAGE MODELS

Over the years attention when assessing quality and performance of comminution processes has been focused on particle size distribution. There are different studies, knowledge and models in coarse comminution that predict the size distribution after crushing. At present, the models in coarse comminution are capable of predicting particle size distribution, capacity and energy. These models have their bases in the empirical model of Whiten (1972), where he presented a model using a population balance model to predict particle size.

Evertsson (2000) presented a model for rock breakage in cone crushers. His work was based on rock breakage characterisation by a piston and die test. This work resulted in a laboratory test procedure for calibrating the crusher model using single and inter-particle breakage. Subsequently, Bengtsson (2009) developed a model capable of predicting particle shape by using bimodal Weibull distribution. His work demonstrates the possibility to use empirical models to model characteristics. The compressive breakage models predict the particle size distribution, however, the existing models are limited by the intrinsic physical properties of the ore, such as the element/mineral concentration or mineral liberation.

### 4.2 LIBERATION MODELS

The evaluation of the mineral liberation is measured in the last step of the comminution process, when all particles are reduced to a certain size in microscale. There are a large number of liberation models that attempt to quantify the degree of liberation by predicting the behaviour of a

particle in a separation process, all of them are focused on fine comminution and consider random fracture.

The liberation models and their mathematical equations are based on the work of A. M. Gaudin (1939) which evaluated the breakage mechanism based on the structure and bonding of the grain. The author described two types of mineral liberation: by comminution and by detachment. Andrew and Mika (1975), Wiegel (1976), Barbery (1987) and King and Schneider (1998) continue with the research and developed a model for the fraction of particles of a certain size that contained less than a determinate fraction of a particular mineral. These models are the basis for today's research, however they have unrealistic assumptions in reference to the grain structure of the minerals. Their work has been followed by others such as Wei and Gay (1999), Gay (2004) and Gay and Latti (2006), who introduced multi-mineral systems, the models of which are valid for different breakage mechanisms. In their work it is suggested that liberation analysis and modelling are beneficial when determining the optimal grind size for the comminution unit, specifically for fine particles, for which they connected the liberation distribution with the texture of the ore and the dispersion rate function. Others works, such as Fandrich et al. (1997), investigated how the mineral liberation was influenced by particle bed breakage. The liberation analysis and modelling are beneficial when determining the optimal grind size for the comminution unit, especially for fine particles, for which they connected the liberation distribution with the texture of the ore and the dispersion rate function.

Moreover, the liberation analysis and prediction is not the only basic fundamental concept that must be understood; it is necessary to understand and assess other concepts, such as fracture process, mineral concentration, breakage mechanisms and the behaviour of different minerals. Fandrich et al. (1997) established different types of breakage of ore, including random and selective breakage. In random breakage, the fracture occurs randomly, without consideration of the different phases present in the ore, allowing the composition of all particles to remain constant. In contrast, in selective or non-random breakage, the fracture is preferential and as a consequence it generates a different concentration in the composition of different size fractions. The work by Mariano et al. (2016) summarized the perspectives on random and non-random breakage. In more recent works, Vizcarra et al. (2010) and Ozcan, Benzer (2013) the mineral liberation was evaluated using different breakage mechanisms.

The liberation of minerals occurs at finer size particles and for this reason all models apply for fine comminution processes. However, during coarse comminution liberation is believed to be minor, although the mineral concentrations in different particle sizes could affect the liberation performance in later stages. Recently, Carrasco et al. (2016) proposed a coarse liberation model based on preferential grade by size response in terms of ranking the response parameter of ores as a function of the particle size distribution and the size reduction process using the Monte Carlo method. Other research such as Bazin et al. (1994), considers that some ores preferentially deport metal into specific size fractions, supporting the development of a mineral concentration model by fitting a third order polynomial. In recent research Hesse et al. (2017), selective breakage was investigated using quantitative microstructural analysis (QMA). The paper contains a detailed analysis on the problems associated with understanding selective breakage, e.g., the mineral geometry and orientation inside the structure.

# 5

## MATERIAL CHARACTERISATION

*The aim of this chapter is to:*

- *Explain the breakage methodology to characterize an ore.*
- *Describe surface analysis of the ore through SEM.*
- *Describe the elemental analysis characterisation.*

### 5.1 MATERIAL CHARACTERISATION BREAKAGE

Evertsson developed a method for characterizing the compressive breakage behaviour of a rock material with the use of a piston and die test. Subsequently Lee (2012) optimized this method. The characterisation method for interparticle breakage (IPB) described by Evertsson (2000) and Lee (2012) was used in this research. A schematic illustration of IPB is shown in Figure 12.

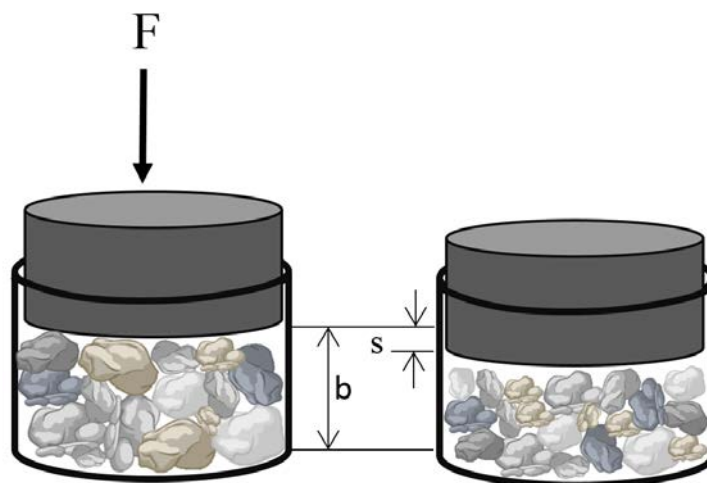


Figure 12. Illustration of Interparticle breakage before and after the compression.

The compressive breakage methodology allows analysis and mechanical characterisation of the rock behaviour in terms of the breakage, particle size distribution and energy consumption when compressive forces are applied. For this analysis, a specific amount of material (350 particles) was placed in a cylinder to apply compression forces. The method suggested the use of monosized particles of 16-19 mm, however, it was not possible to use the same size in all tested ores.

The bed height ( $b$ ) is generated through the thickness occupied by the material. The displacement of the piston ( $s$ ) and the compression ratio ( $s/b$ ) are fixed values. The definition of the compression ratio is presented in equation (1) and it represent the compression by the relation between the initial and final height.

$$S_{\%} = \frac{s}{b} \quad (1)$$

The particle size distribution is obtained by sieving the resulting material. The series of breakage applied in the experiment were conducted according to the test plan developed by Evertsson (2000) and optimized by Lee (2012). The series of breakage simulates the compressive breakage sequence that occurs in a cone crusher. Table 2 shows the test parameters for the mechanical characterisation of the ores. The particle size distribution is obtained by sieving the resulting material. The tests are performed using Instron 400RD hydraulic compression equipment. The technical specifications of the compression testing machine are presented in Table 1.

Table 1. Technical specification of compression testing system

Model	Instron 400RD
Loading capacity	2000 kN
Maximum stroke	76 mm
Load measurement accuracy	± 1.0% of reading down to 4% of frame capacity
Strain measurement accuracy	± 0.5% of reading down to 1/50 of full range
Position measurement accuracy	± 0.5% or 0.13 mm displacement
Position resolution	1 µm
Max test speed	75 mm/min
Data acquisition rate	1 kHz on load and strain
Diameter of die	100 mm



Table 2. Test plans for the material characterizing for interparticle breakage

Test no	Test material	Compression ratio	Chemical Analysis
1	350 particles	0.10	*
2	From no 1	0.10	
3	From no2	0.10	
4	From no 3	0.10	
5	From no 4	0.10	
6	350 particles	0.15	
7	From no 6	0.15	
8	From no 7	0.15	
9	From no 8	0.15	
10	From no 9	0.15	
11	350 particles	0.20	*
12	From no 11	0.20	
13	From no 12	0.20	
14	From no 13	0.20	
15	350 particles	0.25	
16	From no 15	0.25	
17	From no 16	0.25	
18	From no 17	0.25	
19	350 particles	0.30	*
20	From no 19	0.30	
21	350 particles	0.35	
22	From no 21	0.35	

Papers A, B, C and D describe the mechanical characterisation performed in four different ores as well as the results obtained regarding particle size.

## 5.2 MINERALOGICAL CHARACTERISATION

The mineralogical composition of the ore can be analysed by using scanning electron microscopy (SEM) with energy-dispersive X-ray spectroscopy analysis (EDS). The characterisation through this method provides qualitative data of the morphological structure of the ore. This assessment offers information on the minerals present in the sample, liberated particles, composite particles and their mineral associations, grain sizes, difficulty of liberation, cracks and microfractures, among others. The analysis of the particles is focused on minerals related to critical metals and their associations.

The analysis is performed on the product generated from the breakage sequence in the compression breakage test, with a specific compression ratio and size distribution fraction. Three values of compression ratio are analysed. The values are 0.10, 0.20 and 0.30. The selection of the crushed test for mineralogical analysis is shown in Table 2 by the category of chemical analysis.

After the IPB test, the product is divided in nine size fractions as shown in Table 3. The test procedure for mineralogical analysis included 27 samples for the analysis of one ore.

Table 3. Test plan for the mineralogical characterisation

Particle size fraction (mm)	
5.6	>
4.0	5.6
2.0	4.0
1.0	2.0
0.5	1.0
0.25	0.5
0.125	0.25
0.063	0.125
0	0.063

The particles from each size fraction are mounted separately in embedded epoxy resin and polish until a mirror-like surface is achieved. The surface is carbon coated and inspected under the microscope.

The analysis is performed using a Hitachi S-3400N SEM coupled to an Oxford Instrument X-Max EDS, at the Department of Geosciences at the University of Gothenburg. The Oxford Inca software is set up to automatically detect high density minerals by the back-scatter intensity and to analyse the chemistry of each detected grain by EDS.

Papers A, B and C describe the qualitative mineral characterisation performed on three different ores. These papers also address the results obtained regarding minerals, minerals association with respect to the critical metal minerals, fracture of the phases and liberation of the particles, in different size fractions.

### 5.3 ELEMENTAL ANALYSIS CHARACTERISATION

Analytical data of the ore can be acquired using geochemical analysis (bulk chemical analysis - BCA). The geochemical analysis provides quantitative data. This assessment offers analytical data on the distribution and amount of chemical elements in the rock. The rock analysis corresponds to fused bead and acid digestion, then inductively coupled plasma mass spectrometry (ICP-AES) and inductively-coupled mass spectrometry (ICP-MS). ICP-AES is used to measure the content of the major rock-forming elements and minor elements. Major elements are ones whose oxides constitute more than 1% in weight; silica oxide (Si), aluminium (Al), iron (Fe), calcium (Ca), magnesium (Mg), sodium (Na), potassium (K). Minor elements are ones whose oxides constitute between 0.1 and 1% by weight of rock and include titanium (Ti), phosphorus (P), chromium (Cr), and manganese (Mn). ICP-MS quantify the content of trace elements, which are all other metal elements in the periodic table, and occur in concentrations of several parts per million (ppm) and include barium (Ba), gallium (Ga), tantalum (Ta) and tungsten (W) amongst others.

The selection of samples is the same as for the mineral characterisation, meaning that the tested samples are particles from the last interparticle breakage test with a specific compression ratio and size distribution fraction. Nine fractions for three values of compression ratio are analysed. The values are 0.10, 0.20 and 0.30. The selection of the crushed test for mineralogical analysis is shown in Table 2 by the category of chemical analysis. After the IPB test, the product is divided into nine size fractions as shown in Table 3. The test requires a particle size smaller than 70 microns to ensure the quality and validity of the results. Once the product is divided into the fractions, it is necessary to mill all samples to ensure the size requirement of the test. The test procedure for mineralogical analysis includes 27 samples for the analysis of an ore. Additionally, one sample without any compression is tested.

Although, the limitation of this test is the lack of information regarding which minerals the studied element belongs to, also is lacking information on if the particle is being composite or liberated.

The test is performed at the company ALS Geochemistry.

Papers A, B and C describe the quantitative elemental characterisation performed in three different ores. As well as the results obtained regarding major elements, minor and trace elements in different size fractions.

### 5.4 CUMULATIVE DISTRIBUTION OF CONCENTRATION

A cumulative distribution function is going to be the methodology for analysing the geochemical data of the critical metals. The advantage of uses this mathematical technique is that the normalized frequency distribution has no relationship to mass. The data are normalized by dividing each fraction mass by the total mass of all fractions. The cumulative distribution ( $H_o(x)$ ) is the integration of the normalized frequency distribution concentration values ( $\gamma_o$ ) between an interval of fraction size, where ( $c$ ) is the lower size particle limit and ( $d$ ) is the upper size particle limit, as shown in equation (2).

$$H_o(x) = \int_c^d \gamma_o(x) \approx \sum_{i=1}^d \gamma_o(x) \quad (2)$$

### 5.5 EXPERIMENTAL STUDY

#### 5.5.1 Materials

In this study, four different ores were tested: two tungsten ores and two tantalum ores. The studied materials include: one tungsten ore W1 from Spain (granite); one tungsten ore W2 from Austria (felsic gneisses) and one tantalum ore from different sampling, T3 and T4, from Spain (granite). Table 4 shows the distribution of the ores and nomenclature.

Table 4. Test material

Material	Test name	Paper
Tungsten Ore type 1	W1	A
Tungsten Ore type 2	W2	B
Tantalum Ore type 3	T1	C
Tantalum Ore type 4	T2	

All materials were analysed using the procedure for material characterisation presented in previous section. The material characterisation includes interparticle breakage through a series of compressions with three values of compression ratio, followed by the analysis of the product divided in different sizes fractions. These fractions were evaluated with a SEM and geochemical analysis.

Papers A, B and C describe the test method and the results for the three ores in terms of the particle size distribution, minerals and element concentration for different particle size fractions. In the section below some of the obtained results are shown. This method demonstrates that it is possible to analyse different types of ores with different behaviour and properties, and obtain the parameters that can be compared and modelled.

#### 5.5.1.1 Tungsten Ore type 1

W1 is a granite with vein containing scheelite, wolframite, pyrite, arsenopyrite chalcopyrite and cassiterite. The major components of this ore are SiO<sub>2</sub> 74-78 mass%, Al<sub>2</sub>O<sub>3</sub> 12-14 mass%, TiO<sub>2</sub> 0.1-0.2 mass%, Fe<sub>2</sub>O<sub>3</sub> 0.8-1.3 mass%, Na<sub>2</sub>O 2.2-2.9 mass%, CaO 0.4-0.6 mass%, MgO 0.1-0.4 mass%, K<sub>2</sub>O 5.1-6.2 mass% and P<sub>2</sub>O<sub>5</sub> 0.3-0.4 mass%. The main minerals phases are quartz, albite, K-feldspar and muscovite (Alfonso et al. 2016).

Figure 13 and Figure 14 show the product size distribution after a compression breakage with 0.20 of compression ratio.

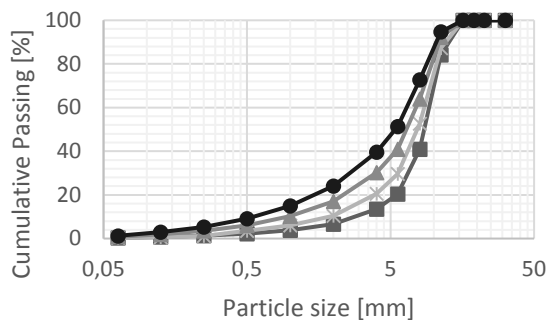


Figure 13. Product particle size distributions for the IPB 20% test series (cumulative) for W1.

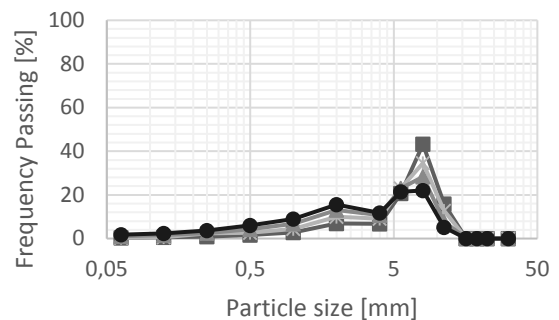


Figure 14. Product particle size distributions for the IPB 20% test series (frequency) for W1.

After the compression, size fractions of the product were analysed in SEM. W1 is a tungsten ore with two main sources of this metal, scheelite and wolframite. In backscattered images, the heavy minerals are recognised by their lightness and brightness. As tungsten is a dense element, it is possible to recognise the particles with the content of these minerals (scheelite or wolframite), because they are lighter and brighter compared with other minerals.

Scheelite was found together with muscovite, K-feldspar, quartz, apatite and albite. Normally the surrounded minerals show the presence of fractures or micro-fractures. The scheelite particles present a simple locking texture, which allows liberation due to the rectilinear or gently curved boundaries. Figure 15 to Figure 18 show composite particles of scheelite, and Figure 19 and Figure 20 shown liberated particles of scheelite.

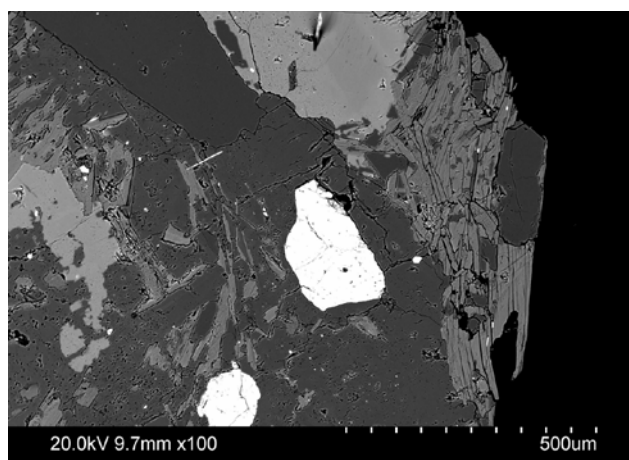


Figure 15. Backscattered electron image of a composite particle of W1 after IPB at 0.30. The particle is embedded in by scheelite and albite.

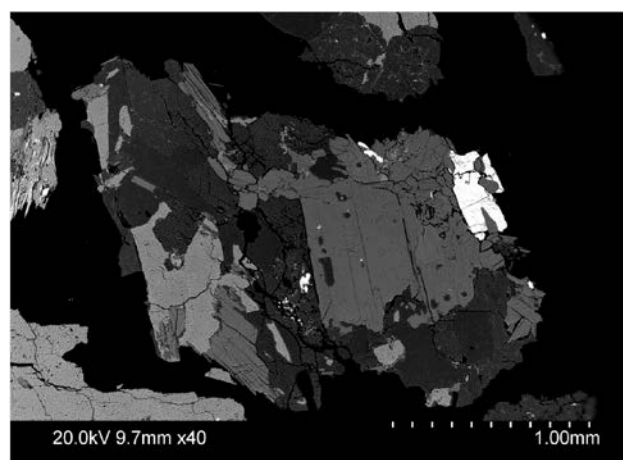


Figure 16. Backscattered electron image of a composite particle of W1 after IPB at 0.10. The particle is embedded in by scheelite and K-feldspar.

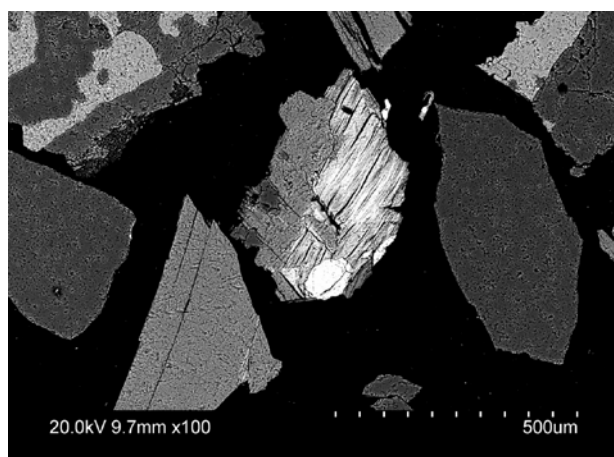


Figure 17. Backscattered electron image of a composite particle of W1 after IPB at 0.10. The particle is embedded in by scheelite and apatite and muscovite.

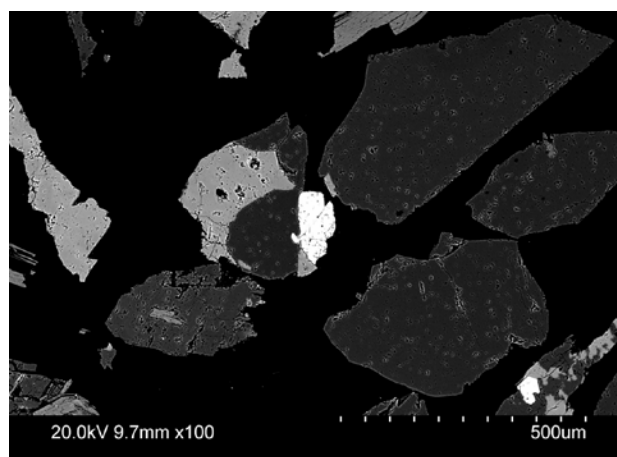


Figure 18. Backscattered electron image of a composite particle of W1 after IPB at 0.30. The particle is embedded in by scheelite in apatite.

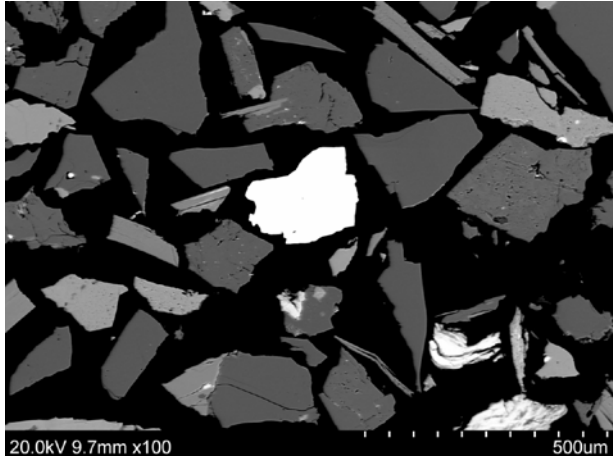


Figure 19. Backscattered electron image of a scheelite liberated particle of W2 after IPB at 0.10.

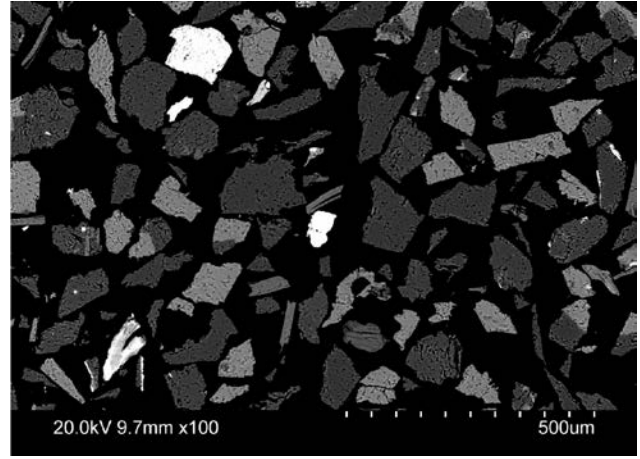


Figure 20. Backscattered electron image of a scheelite liberated particle of W2 after IPB at 0.20.

The other main ore mineral in W1 is wolframite. It was observed that the wolframite is mixed with other minerals rich in iron such as iron oxide or iron silicates. Figure 21 to Figure 24 show particles with wolframite in association with other minerals, common associations are with K-feldspar, quartz, albite, iron silicates, muscovite, and biotite. Wolframite particles are different to the scheelite. The wolframite is not an entire grain, on the contrary it is an extension that crosses different minerals and grains, like veins or branches that are occupying a crevice or fissure in the particle. These textures correspond to a vein-like network type, which suggests that a complete liberation would be fairly difficult. Generally, when one particle has wolframite in it, all the particles contain small proportions of it blended in forms of small grains or veins that cross the particle. The liberation of wolframite is difficult and will require the reduction of the particle into very small sizes in order to be able to separate the different minerals.

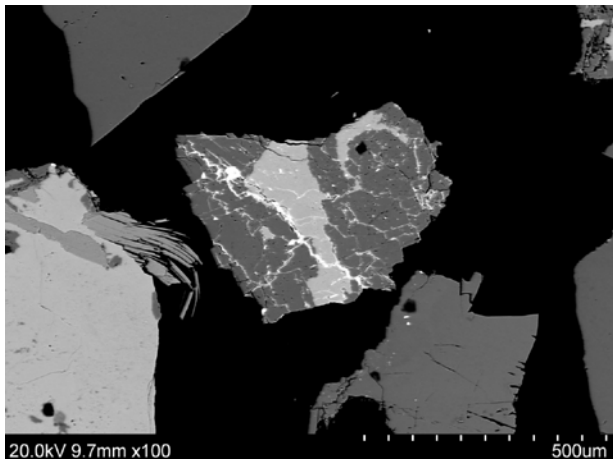


Figure 21. Backscattered electron image of a composite particle of W1 after IPB at 0.10. The particle is embedded in by wolframite and quartz and albite.

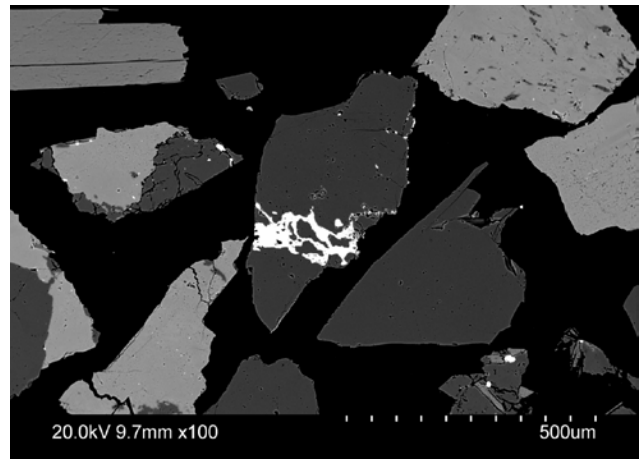


Figure 22. Backscattered electron image of a composite particle of W1 after IPB at 0.30. The particle is embedded in by wolframite and quartz.

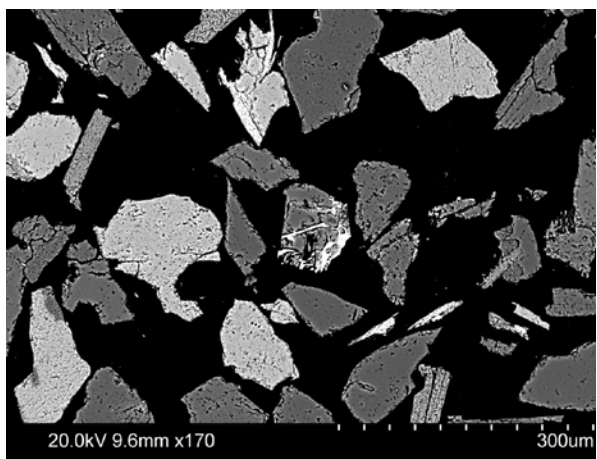


Figure 23. Backscattered electron image of a composite particle of W1 after IPB at 0.10. The particle is embedded in by wolframite and quartz and albite.

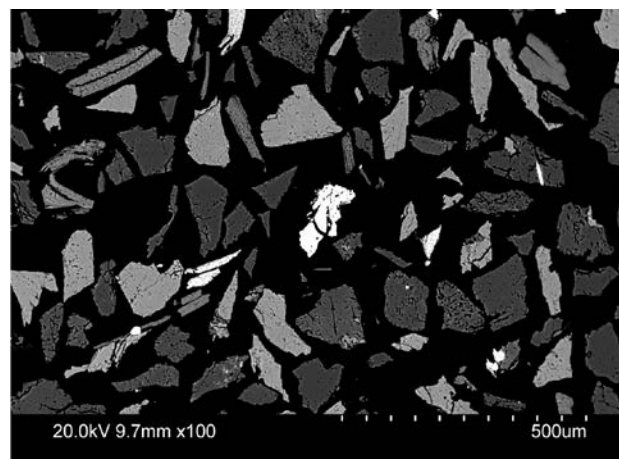


Figure 24. Backscattered electron image of a wolframite liberated particle of W2 after IPB at 0.10.

The results for the elemental composition of the ore are shown in Table 5 after a breakage with a compression ratio of 0.30. Figure 25 shows the summary of the content of tungsten in each size fraction. According to the equation (2), the normalized cumulative concentration is shown in Figure 26.

Table 5. Summary of representative composition of W1 ore after compression breakage at a compression ratio of 0.30

Size fraction	Critical Metals		Major elements							
	Ta	W	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	Total
mm	ppm	ppm	%	%	%	%	%	%	%	%
5.6	2.2	103	74.7	13.0	1.42	0.50	0.32	2.73	5.21	99.49
4	2.0	193	76.2	13.0	1.37	0.43	0.28	2.57	5.68	101.10
2	1.0	20	76.6	13.3	1.17	0.42	0.23	2.43	5.62	101.53
1	1.9	57	74.9	12.9	1.15	0.40	0.19	2.66	5.63	99.15
0.5	2.5	110	74.0	14.2	1.12	0.38	0.24	2.56	6.16	100.11
0.25	2.8	97	73.9	14.8	1.24	0.45	0.26	2.81	6.27	101.25
0.125	2.6	483	71.8	14.5	1.28	0.55	0.25	3.07	6.00	99.05
0.063	2.5	316	71.8	15.4	1.27	0.85	0.25	3.64	6.13	101.15
rest	2.8	456	69.7	15.7	1.32	1.12	0.20	4.09	6.17	101.02

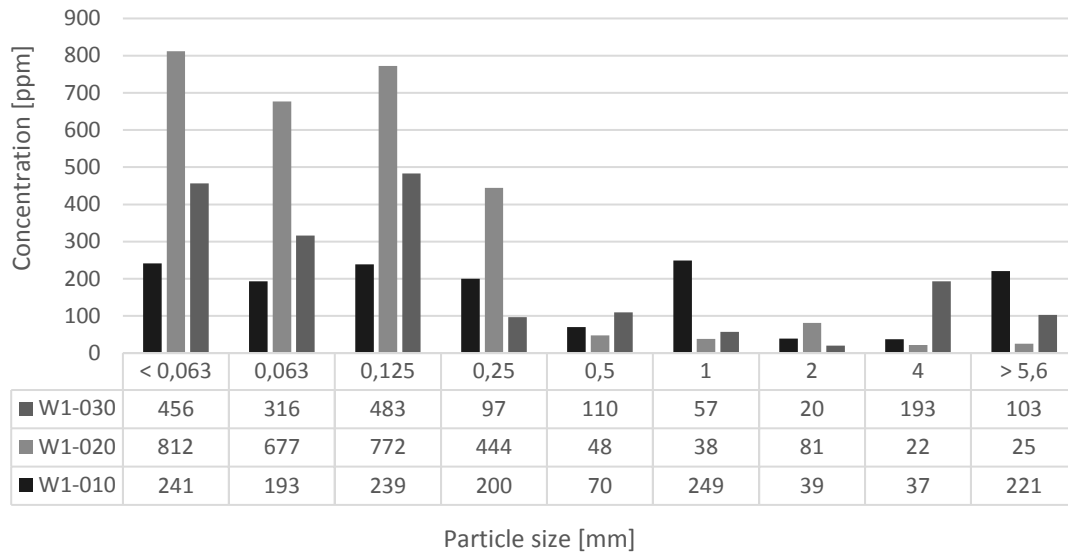


Figure 25. Measured data from geochemical analysis for W1.

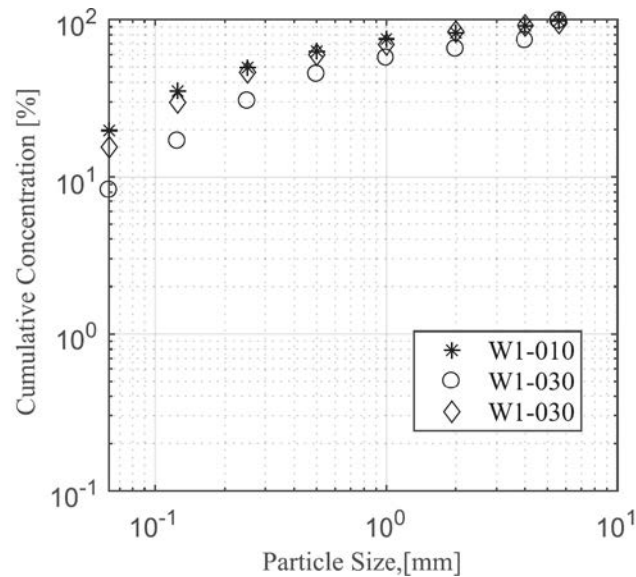


Figure 26. Cumulative normalized concentration for ore W1

#### 5.5.1.2 Tungsten Ore type 2

W2 comes from a scheelite deposit of amphibolites and felsic gneisses. The grade of the deposit is 0.3 mass% located in quartz veins. The main constituents are hornblendites and coarse grained amphibolites. The major minerals present in the ore are amphibole, biotite, quartz, plagioclase, scheelite and some pyrite, pyrrhotite and limonite are present as accessory minerals. The major components of this ore are SiO<sub>2</sub> 49-78 mass%, Al<sub>2</sub>O<sub>3</sub> 4-19 mass%, Fe<sub>2</sub>O<sub>3</sub> 0.6-12 mass%,



Na<sub>2</sub>O 0.3-6 mass%, CaO 3.5-12.5 mass%, MgO 5-38 mass% and K<sub>2</sub>O 0.8-2.8 mass% (Thalhammer 1987).

Figure 27 and Figure 28 show the product size distribution after a compression breakage with 0.20 compression ratio.

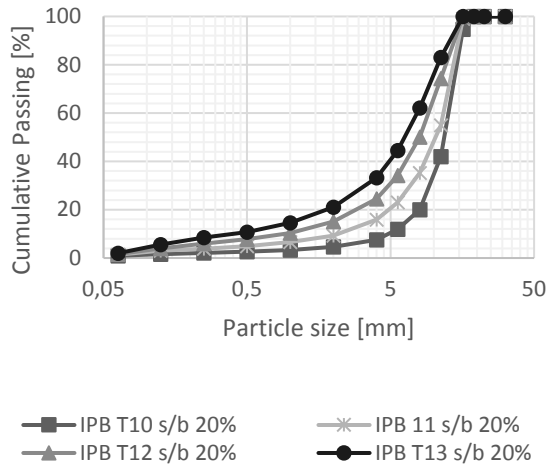


Figure 27. Product particle size distributions for the IPB 20% test series (cumulative) for W2.

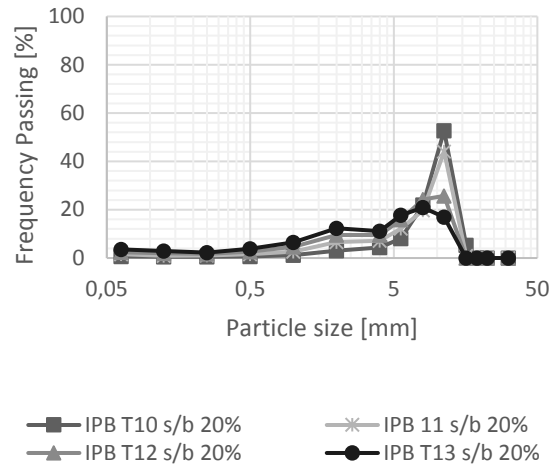


Figure 28. Product particle size distributions for the IPB 20% test series (frequency) for W2.

After the compression, size fractions of the product were analysed in SEM. W2 is dominated by major minerals such as quartz, albite, biotite, k-feldspar, chlorite, anorthite, hornblende, calcite and fluorite. The main ore mineral is scheelite. Generally, scheelite was found in association with biotite, K-feldspar, quartz, apatite and albite, as Figure 29 and Figure 30 show. Figure 31 and Figure 32 show liberated scheelite particles, it was common to find liberated scheelite at finer particle size fraction, between 0.063 and 0.125 mm at any compression ratio.

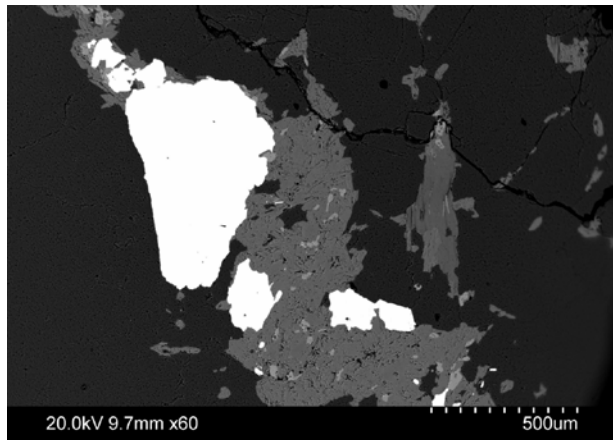


Figure 29. Backscattered electron image of a composite particle of W2 after IPB at 0.30. The particle is embedded in by scheelite and quartz, apatite, chlorite, hornblende and titanite.

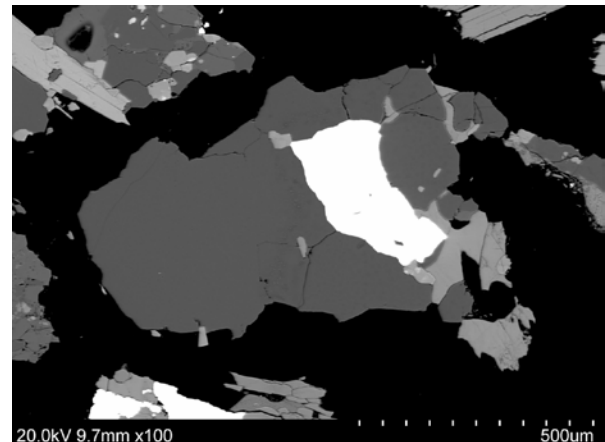


Figure 30. Backscattered electron image of a composite particle of W2 after IPB at 0.10. The particle is embedded in by scheelite and quartz, hornblende and calcite.

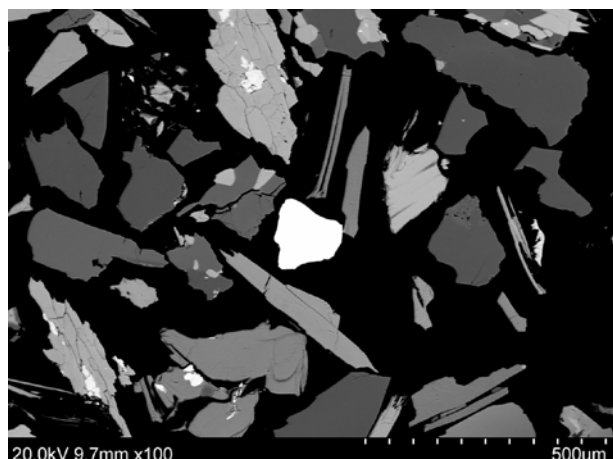


Figure 31. Backscattered electron image of a scheelite liberated particle of W2 after IPB at 0.10.



Figure 32. Backscattered electron image of a scheelite liberated particle of W2 after IPB at 0.20.

The geochemical analysis results for the tungsten content are presented in Table 6. Figure 33 shows the summary of the content of tungsten in each size fraction. The normalized cumulative concentration is shown in Figure 34.

Table 6. Summary of representative composition of W2 ore after compression breakage at a compression ratio of 0.30.

Size fraction	Critical Metals		Major elements							
	Ta	W	SiO2	Al2O3	Fe2O3	CaO	MgO	Na2O	K2O	Total
mm	ppm	ppm	%	%	%	%	%	%	%	%
5.6	1.0	590	60.0	12.1	7.51	7.96	5.24	2.76	1.08	9.,52
4	0.7	408	57.1	13.2	8.47	9.23	6.21	2.87	0.96	100.48
2	0.9	965	59.7	12.5	8.19	8.30	6.35	2.73	1.01	101.09
1	0.9	1750	56.7	12.7	8.54	8.57	6.55	2.79	1.02	99.36
0,5	0.8	1170	57.6	12.7	7.90	7.85	6.27	2.86	1.17	98.62
0.25	1.1	1660	59.7	12.5	7.53	6.85	5.92	2.88	1.65	99.68
0.125	1.5	2010	59.7	12.4	7.75	7.07	6.32	2.59	2.01	100.46
0.063	1.3	1740	52.6	12.1	9.78	10.10	8.22	2.12	1.15	98.86
rest	1.4	1890	50.9	12.1	10.65	11.65	8.70	1.83	0.73	99.69

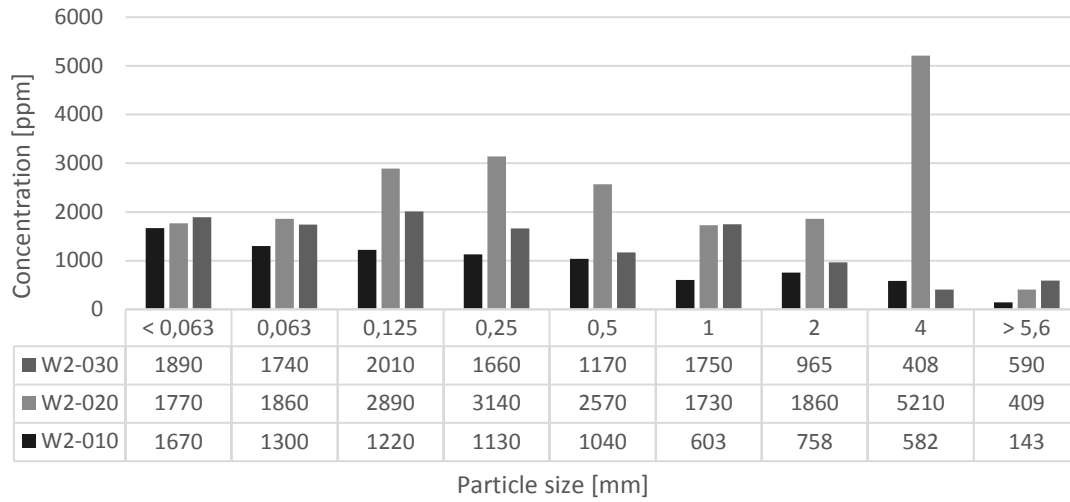


Figure 33. Measured data from geochemical analysis for W2.

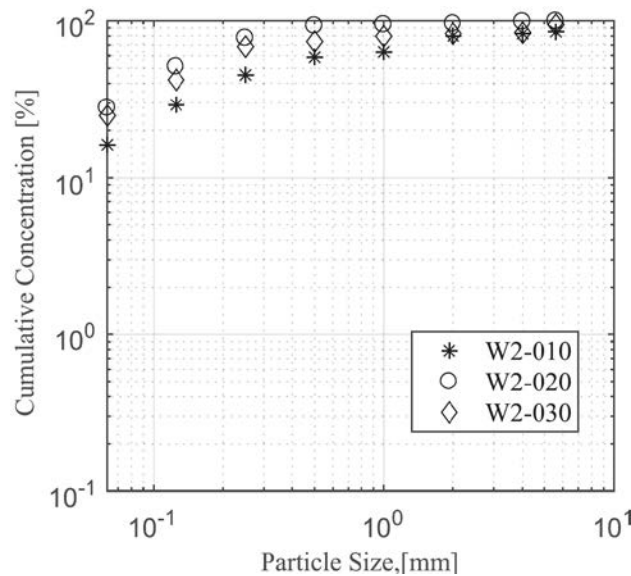


Figure 34. Cumulative normalized concentration for ore W2.

### 5.5.1.3 Tantalum Ore type 3

T3 and T4 ore is a granite with veins rich in quartz, cassiterite and columbite group minerals. The essential minerals are plagioclase, quartz, potassium, K-feldspar and white micas. Accessory minerals are present such as garnet, cassiterite, apatite, monazite, zircon, columbite-tantalite and uraninite. The major components of this ore are  $\text{SiO}_2$  70-76 mass%,  $\text{Al}_2\text{O}_3$  14.2-16.6 mass%,  $\text{Fe}_2\text{O}_3$  0.4-1.12 mass%,  $\text{Na}_2\text{O}$  4.3-6.7 mass%,  $\text{CaO}$  0.1-0.15 mass%,  $\text{MgO}$  0.01-0.03 mass% and  $\text{K}_2\text{O}$  3.35-4.1 mass% (Llorens González et al. 2017, López et al. 2018, López-Moro et al. 2017).

Figure 35 and Figure 36 show the product size distribution after a compression breakage with 0.20 compression ratio. There is an accumulation of particles in the size ranges of 1–2 mm and 2–4 mm, represented by the frequency figure. The accumulation of particles is due to the mineralogy of the ore.

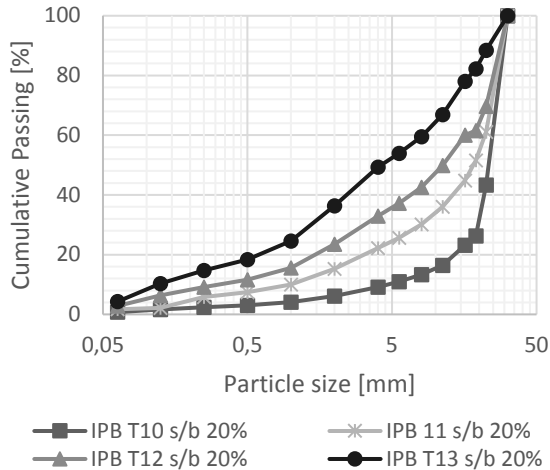


Figure 35. Product particle size distributions for the IPB 20% test series (cumulative) for T3.

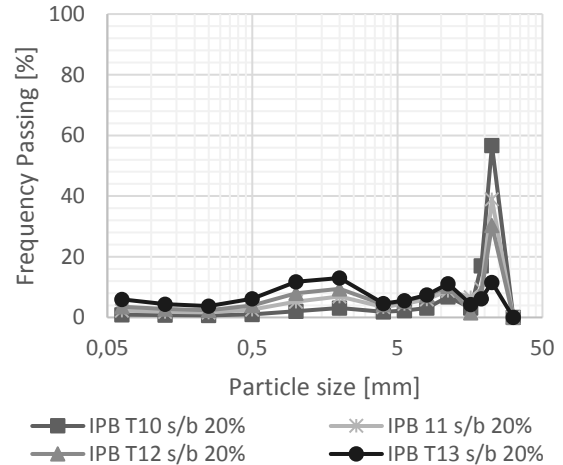


Figure 36. Product particle size distributions for the IPB 20% test series (frequency) for T3.

After the compression, size fractions of the product were analysed in SEM. The main ore minerals in T3 and T4 are cassiterite, tantalite and columbite. The dominated minerals are quartz, albite, muscovite and K-feldspar, as Figure 37 and Figure 38. Liberated particles of tantalite were found on size particle of 0.125 and 0.063 mm at any compression ratio, as Figure 39 shows.

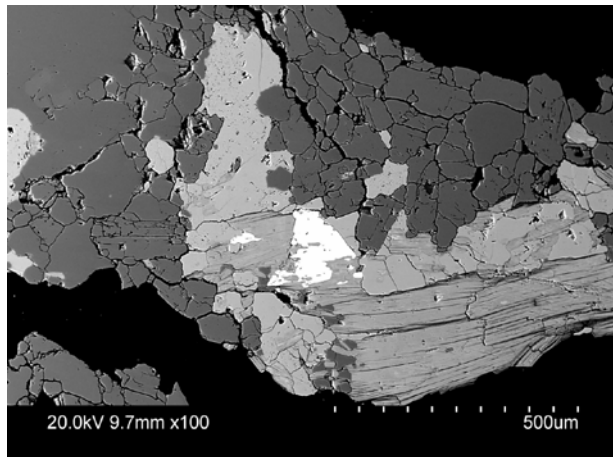


Figure 37. Backscattered electron image of a composite particle of T3 after IPB at 0.10. The particle is embedded in by tantalite, K-feldspar, muscovite and quartz.

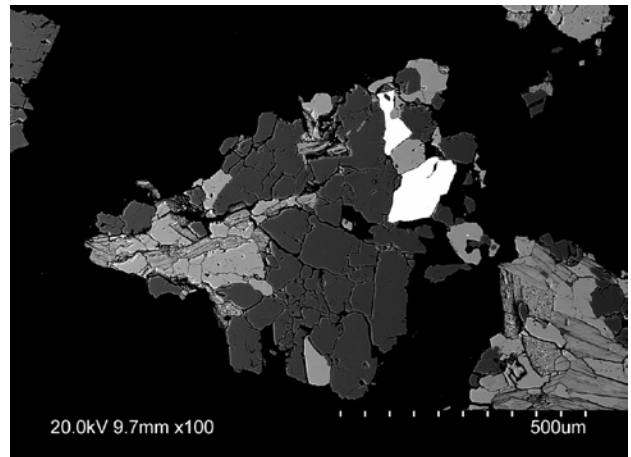


Figure 38. Backscattered electron image of a composite particle of T3 after IPB at 0.10. The particle is embedded in by tantalite, cassiterite, K-feldspar and quartz.

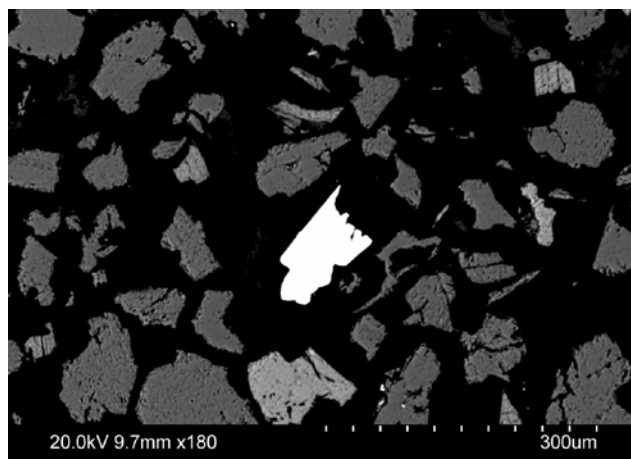


Figure 39. Backscattered electron image of a liberated tantalite particle of T3 after IPB at 0.10.

The geochemical analysis results for element content for T3 are presented in Table 7. Figure 40 shows the summary of the content of tantalum in each size fraction. According to the equation (2), the normalized cumulative concentration is shown in Figure 41.

Table 7. Summary of representative composition of T3 ore after compression breakage at a compression ratio of 0.30.

Size fraction	Critical Metals		Major elements							
	Ta	W	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	Total
mm	ppm	ppm	%	%	%	%	%	%	%	%
5.6	116.0	2	74.7	16.4	0.56	0.10	0.02	5.60	3.44	101.88
4	82.8	2	75.2	15.1	0.56	0.07	0.01	5.01	3.39	100.27
2	71.4	2	79.7	13.2	0.52	0.08	0.02	4.30	2.80	101.44
1	76.5	3	75.5	13.6	0.81	0.07	0.02	3.81	3.38	98.14
0,5	135.5	4	69.1	18.2	0.53	0.07	0.02	3.95	6.04	99.15
0.25	179.0	4	68.7	19.3	0.51	0.10	0.02	4.72	5.85	100.63
0.125	132.0	2	68.2	19.3	0.26	0.13	0.02	7.63	3.02	99.51
0.063	80.9	2	66.8	19.7	0.23	0.13	0.02	8.19	2.54	98.52
rest	79.8	5	67.2	20.3	0.32	0.13	0.04	7.70	2.87	99.95

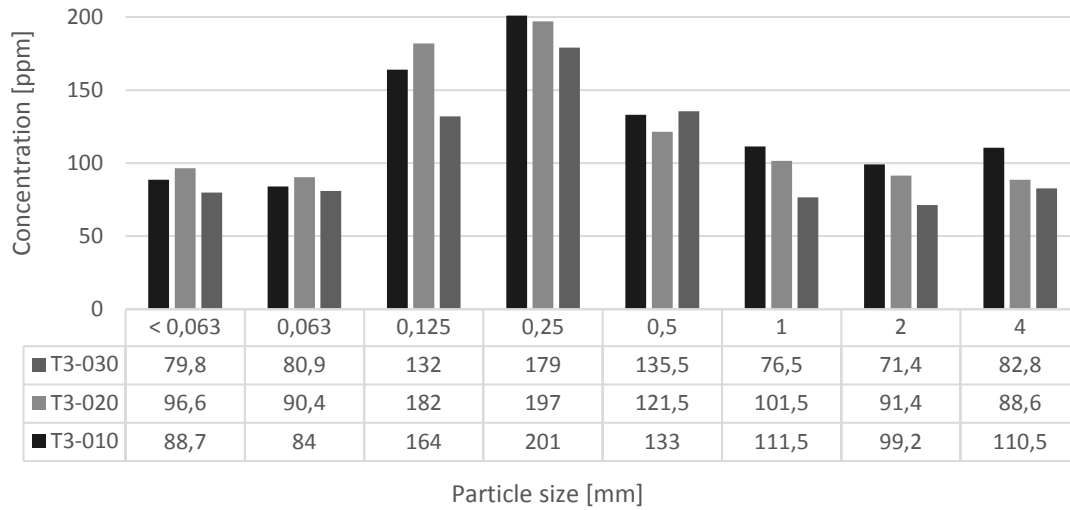


Figure 40. Measured data from geochemical analysis for T3.

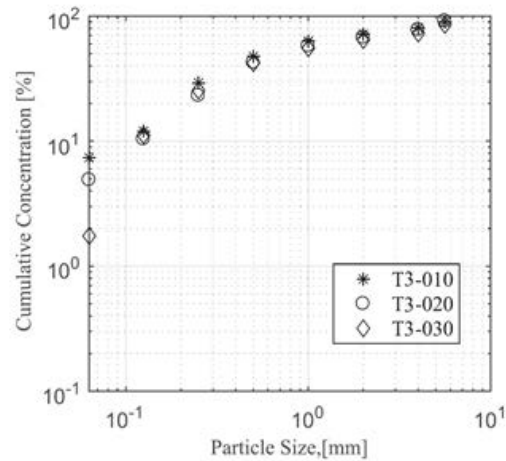


Figure 41. Cumulative normalized concentration for ore T3.

#### 5.5.1.4 Tantalum Ore type 4

T4 and T3 are the same ore, with different sampling. The differences in the sampling is related to the location from where they were selected. Figure 42 and Figure 43 show the product size distribution after a compression breakage with 0.20 compression ratio. There is an accumulation of particles in the size ranges of 1–2 mm and 2–4 mm, represented by the frequency figure. The accumulation of particles is due to the mineralogy of the ore.

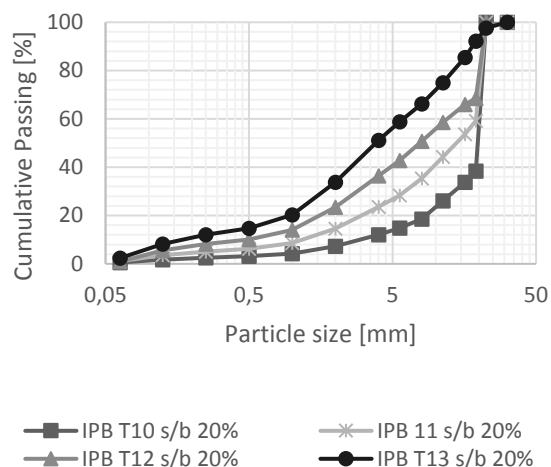


Figure 42. Product particle size distributions for the IPB 20% test series (cumulative) for T4.

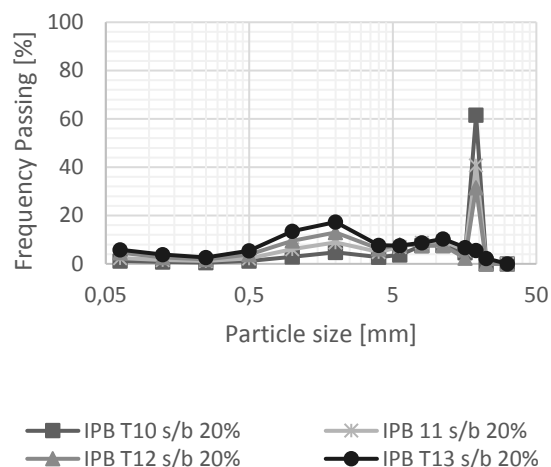


Figure 43. Product particle size distributions for the IPB 20% test series (frequency) for T4.

Figure 44, Figure 45 and Figure 46 show composite particles. Liberated particles of tantalite were found at fractions of 0.125 and 0.063 mm at any compression ratio, as shown in Figure 47.

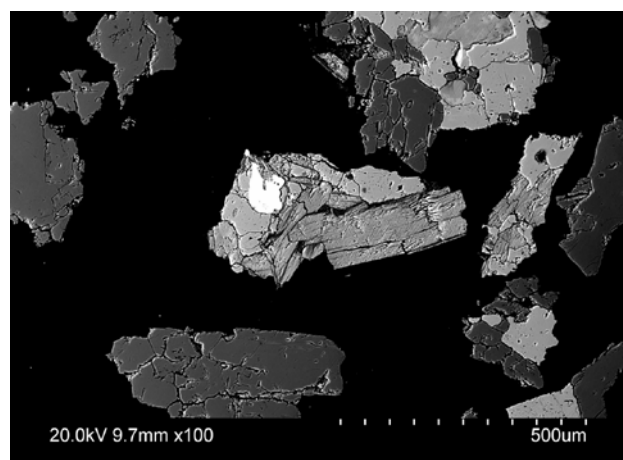


Figure 44. Backscattered electron image of a composite particle of T4 after IPB at 0.10. The particle is embedded in by tantalite, K-feldspar and muscovite.

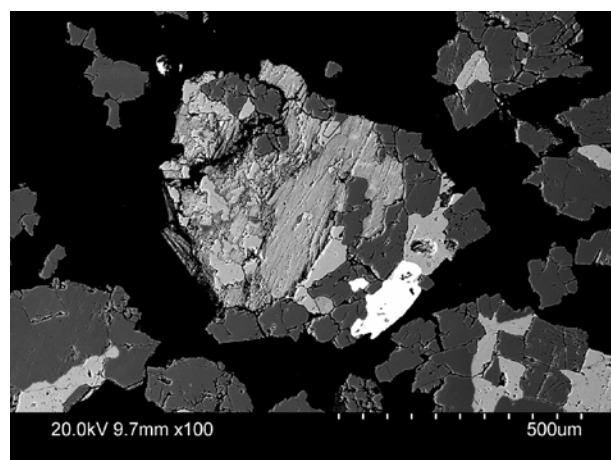


Figure 45. Backscattered electron image of a composite particle of T4 after IPB at 0.10. The particle is embedded in by tantalite, K-feldspar and albite.

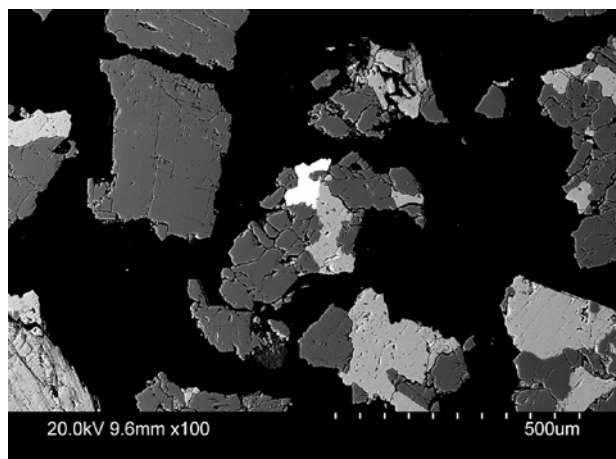


Figure 46. Backscattered electron image of a composite particle of T4 after IPB at 0.20. The particle is embedded in by tantalite, albite and K-feldspar.

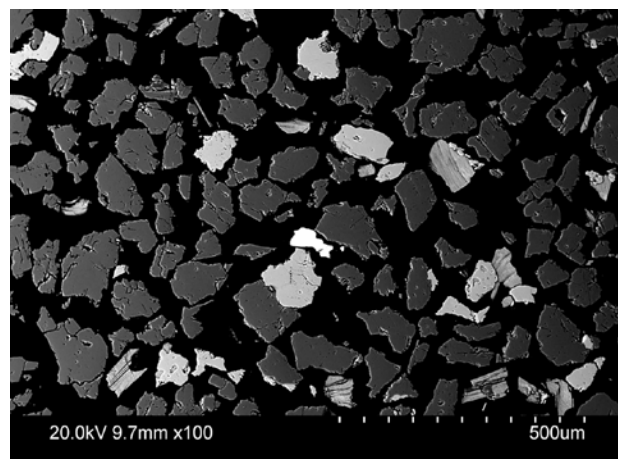


Figure 47. Backscattered electron image of a tantalite liberated particle of T4 after IPB at 0.10.

The geochemical analysis results for element content for T4 are presented in Table 8. Figure 48 shows the summary of the content of tantalum in each size fraction. The normalized cumulative concentrations is shown in Figure 49.

Table 8. Summary of representative composition of T4 ore after compression breakage at a compression ratio 0.30.

Size fraction	Critical Metals		Major elements							
	Ta	W	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	Total
mm	ppm	ppm	%	%	%	%	%	%	%	%
5.6	144.0	4	70.2	18.8	0.81	0.08	0.07	4.58	3.93	100.61
4	125.0	8	67.0	20.9	1.23	0.04	0.11	3.99	4.11	100.65
2	82.3	2	75.9	15.6	0.60	0.07	0.02	4.10	3.16	101.09
1	89.4	5	72.1	16.8	1.18	0.04	0.04	3.09	4.04	99.72
0,5	132.0	5	65.5	21.8	0.91	0.05	0.06	2.83	6.15	100.45
0.25	166.5	8	59.4	24.7	1.33	0.03	0.12	2.66	6.96	99.70
0.125	141.5	5	64.0	23.6	0.72	0.10	0.08	5.85	3.47	101.36
0.063	93.5	9	62.4	23.6	0.72	0.10	0.05	7.19	2.54	100.17
rest	71.3	3	58.4	27.4	0.50	0.13	0.06	5.29	2.10	100.69



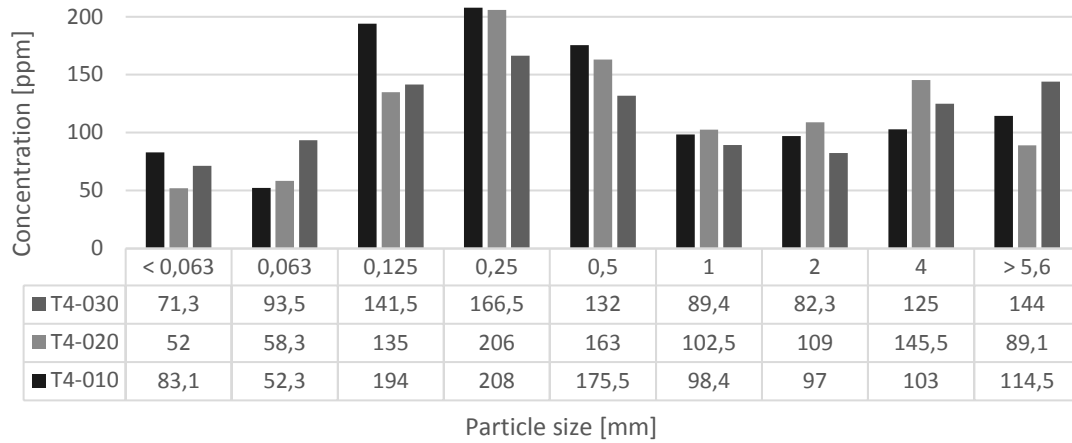


Figure 48. Measured data from geochemical analysis for T4.

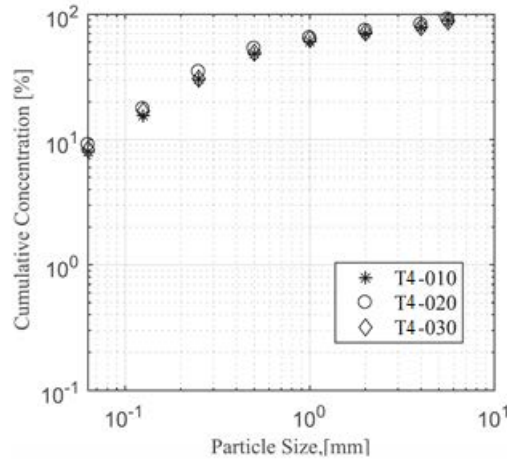


Figure 49. Cumulative normalized concentration for ore T4.

### 5.5.2 Discussion

Papers A, B and C demonstrate three completely different types of ores, with different particle size distribution after compressive breakage, different mineralogical composition and different chemical composition. Nonetheless this testing methodology, allows the interpretation and evaluation of different behaviour and properties, acquiring similar parameters between them that could be compared and modelled.

By analysing the four ores and their behaviour, common results were found. After the compression breakage, the particle size distribution is dependent on the compression ratio and the number of cycles. Higher compression ratio generates more reduction in the particle size, which in turn results in more particles in the fine fraction. Likewise, a larger number of cycles produces more size reduction as Figure 50 suggests.

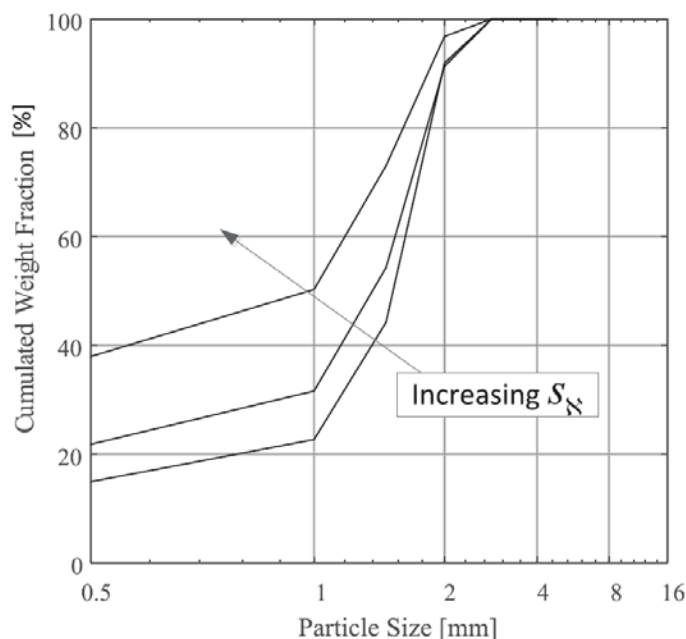


Figure 50. Compression ratio and its influence on size reduction.

The surface analysis with SEM gives qualitative information on the minerals that constitute the particle. With this method it is possible to identify how minerals fracture and how simple the liberation is. In the case of W1 the use of SEM is extremely important due to the fact that this ore has two minerals with tungsten, scheelite and wolframite. Even when both minerals contain tungsten, the texture is different, causing the necessity of using different techniques for the comminution of both minerals. Wolframite require high reduction, as was observed in the SEM pictures, while scheelite is easily liberated with normal comminution process.

Likewise, this method allows the identification of other minerals related to the rare metal mineral. For example, for ores W2, T3 and T4, a strong relation was observed between micas and critical metals. The tantalite and scheelite was generally sitting together with the muscovite, biotite or other micas. Micas are a brittle mineral due to its crystalline structure, so less energy is required to break it and to achieve the size reduction of its particles. This type of mineral rupture generated finer particles and good liberation during the early stages of the breakage process. As the critical mineral was sitting together with the micas, the critical mineral is going to be easily liberated or located in finer size fractions. The coarser fraction is going to be formed mainly by the hardest minerals such as quartz and K-feldspar. Figure 51 suggests the theoretical distribution of the minerals after the size reduction.

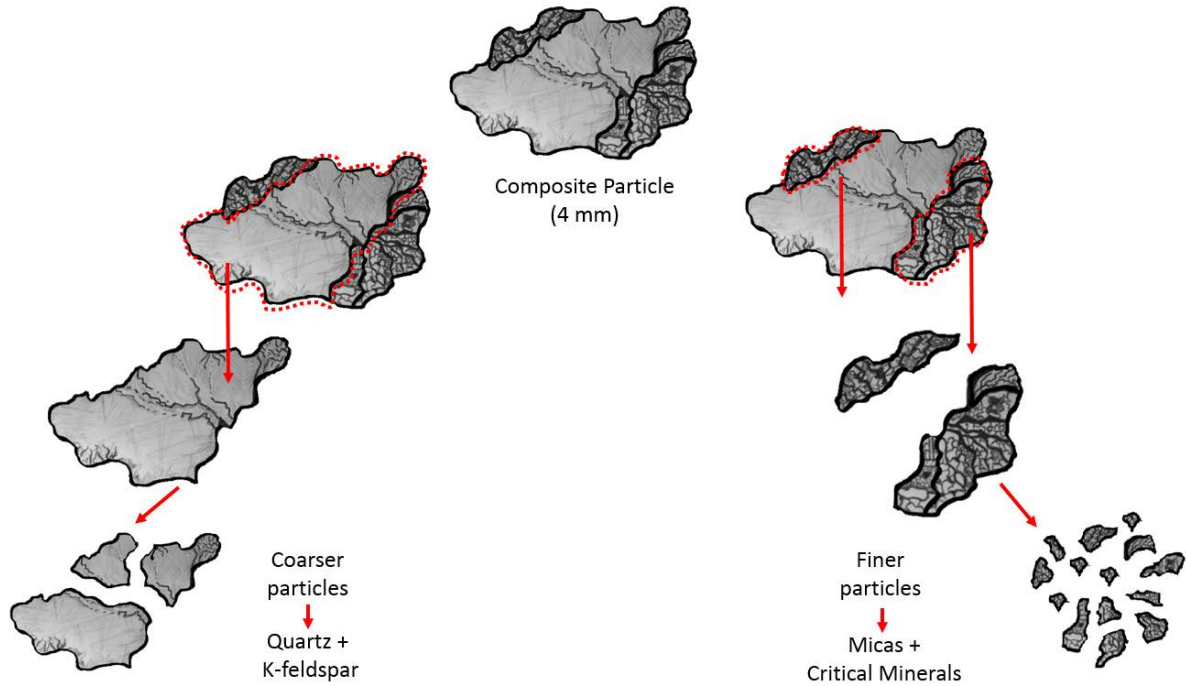


Figure 51. Schematic model of theoretical distribution of minerals.

In regards to the element concentration, major, minor and traces elements, exhibit a smooth variation in concentration with respect to particle size fraction, this means that the distribution cannot be defined as unimodal. This fact is an indication that when the breakage occurs, the minerals are broken differently and into different sizes. Nevertheless, the element concentration did not show any relation to the compression ratio. No relation between the variation in the element content and the value of compression ratio at different sizes was evident in any of the ores.

Another general observation was that some major elements, specifically silica and aluminium presented smaller variations in each size fractions. Quartz is the main mineral that forms these ores and its volume could represent between 45 and 80% of the total volume. For large amounts of mineral content, it would be expected that its concentration would present low variation in different size fractions.

As was mentioned before, the element concentration is a quantitative method capable of measuring the amount of an element in the sample. However, this value cannot be directly related to the minerals or the amount of, minerals, in particular when two or more minerals contribute to the same element. Likewise, this analytical data is not related to the liberation of particles.



# 6

## MODELLING

*The aim of this chapter is to:*

- *Present the particle size model and its calibration.*
- *Describe the concentration model.*

### 6.1 MODELLING PARTICLE SIZE

The compressive reduction process consists of two basic mechanisms: selection of the particles for breakage (S) and breakage of the particles (B). The presented model is based on the work of Evertsson (2000). The model predicts the size distribution as a function of compressing forces and material characteristics. In his work the inter-particle breakage (IPB) test was modelled using a nonlinear regression model following the principle presented by Whiten (1972), and is based on the population balance model presented by Broadbent and Callcott (1956). The model is presented in Figure 52. The selection, S, represents the probability of a particle being broken, and breakage, B, represents the way a particle is broken.

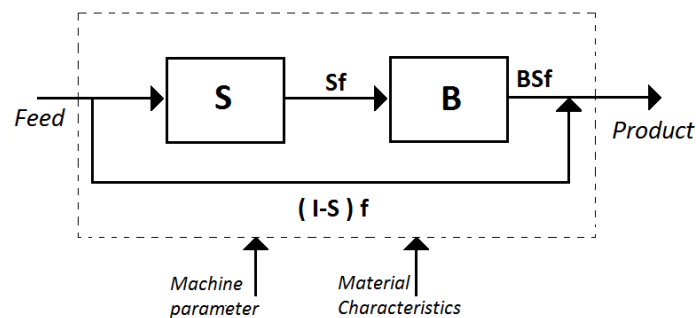


Figure 52. Simplified model of a reduction cycle

The breakage function describes how a compressed particle breaks into smaller fragments. The outcome of this function is the resulting particle size distribution, which is expressed cumulatively. The relative breakage of particles is dependent only on the applied compression ratio  $s_{s/b}$ . According to Evertsson, the breakage function for interparticle breakage is represented in equation (3).

$$B(x_{s/b}, S_{s/b}) = \left(1 - (\alpha_3 + \alpha_4(S_{s/b}))\right) x_{s/b}^{\alpha_1 + \alpha_2(S_{s/b})} + (\alpha_3 + \alpha_4(S_{s/b})) x_{s/b} \quad (3)$$

Here  $x_{s/b}$  is a vector of particle sizes relative to the initial particle size  $x$ . The values of the fitted constants  $\alpha_i$  correspond to the experimental data obtained through the compression tests.  $x_{s/b}$  represents a vector of relative particle size, and is defined in the equation (4)

$$x_{s/b} = \frac{\log_2\left(\frac{x}{x_{\min}}\right)}{\log_2\left(\frac{x_o}{x_{\min}}\right)} \quad (4)$$

During the compression breakage, not all particles will be reduced in size due to the distribution of forces. The selection function describes the possibility for a particle to break, and is assumed to be equal or equivalent for all the particles regardless of their size. This means that the selection will only be dependent on the compression ratio ( $s_{s/b}$ ) and the feed size distribution ( $\sigma_{s/b}$ )

The expression to describe the selection function for interparticle breakage is presented in equation (5).

$$S(S_{s/b}, \sigma_{s/b}) = a_1 S_{s/b}^2 \sigma_{s/b}^2 + a_2 S_{s/b}^2 \sigma_{s/b} + a_3 S_{s/b}^2 + a_4 S_{s/b} \sigma_{s/b}^2 + a_5 S_{s/b} \sigma_{s/b} + a_6 S_{s/b} + a_7 \sigma_{s/b}^2 + a_8 \sigma_{s/b} + a_9 \quad (5)$$

$$\sigma_{s/b} = \frac{\sigma}{x} \quad (6)$$

The values of the fitted constants  $a_i$  correspond to the experimental data obtained through the compression tests.

### 6.1.1 Calibration of the particle size reduction model

The breakage function was fitted to the particle size distribution obtained from laboratory testing using different values of compression ratios (compression ratio: 10, 15, 20, 25, 30 and 35%). Figure 53 to Figure 55 show the data obtained for W2 ore for compression ratio of 10, 20 and 30%.

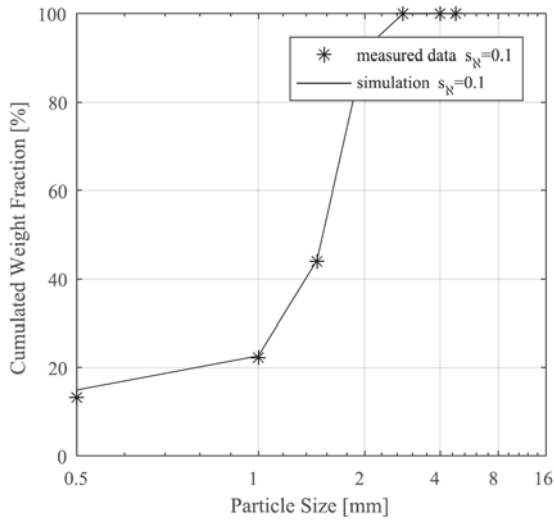


Figure 53. Comparison between the simulated and measured data of the particle size distribution for W2 at 10% of compression ratio.

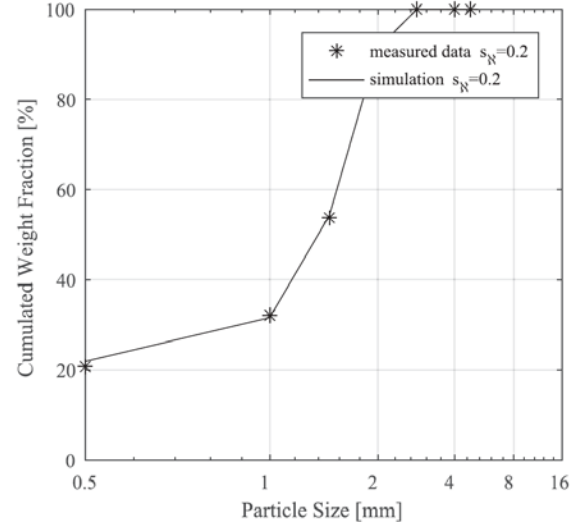


Figure 54. Comparison between the simulated and measured data of the particle size distribution for W2 at 20% of compression ratio.

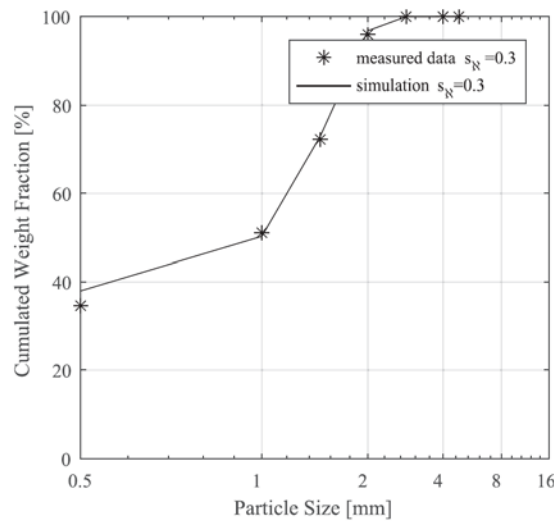


Figure 55. Comparison between the simulated and measured data of the particle size distribution for W2 at 30% of compression ratio.

As can be seen in the figures, the model correlates well with the measured data. Also, from the results it is possible to conclude that the size reduction is affected by the compression ratio. At higher compression ratio the reduction will be higher.

The same validation process was applied for all ores. The fitted breakage function for each ore is described in Paper B and Paper C.

## 6.2 MODELLING OF CONCENTRATION

As previously stated, during coarse comminution the available models are able to predict size reduction, size distribution, capacity and energy composition. This chapter aims to increase the resolution of the crusher models, considering or adding new factors that haven't been considered previously. One important concept is the liberation, however, in coarse

comminution the liberation is minor due to the sizes of the particles. During crushing processes the particle size occurs in millimetres, when in general the liberation occurs in microns range. This explains the reason that it is complex to include liberation concepts in coarse comminution models. Despite this after the characterisation of different ores it was observed that the distribution of certain elements are not homogeneous or constant in each size fraction, this opens up new possibilities to model the distribution of the critical elements after compressive breakage. Paper D describes the methodology to develop a model capable of predicting the critical element concentration.

### 6.2.1 Development of the model

The Weibull distribution has been used for modelling reliability distributions and it is well known for its flexibility. The Weibull distribution is shown in equation (7). The parameters  $\lambda$  and  $v$  are calibration constants that can be determined by the Weibull diagram. As shown in Figure 56 by working mathematically with equation (2) it is possible to obtain the following equations. Equation (8) is obtained by rearranging the terms in the equation, whereas equation (9) is obtained applying the logarithm twice in the equation.

$$F(x) = 1 - e^{-\left(\frac{x}{\lambda}\right)^v} \quad (7)$$

$$\frac{1}{1 - F(x)} = e^{\left(\frac{x}{\lambda}\right)^v} \quad (8)$$

$$\ln\left(\ln\left(\frac{1}{1 - F(x)}\right)\right) = v \ln(x) - v \ln(\lambda) \quad (9)$$

For a better visualization, equation (9) can be transformed into equation (10) to (13).

$$Y = \ln\left(\ln\left(\frac{1}{1 - F(x)}\right)\right) \quad (10)$$

$$X = \ln(x) \quad (11)$$

$$C = -v \ln(\lambda) \quad (12)$$

$$Y = vX + C \quad (13)$$



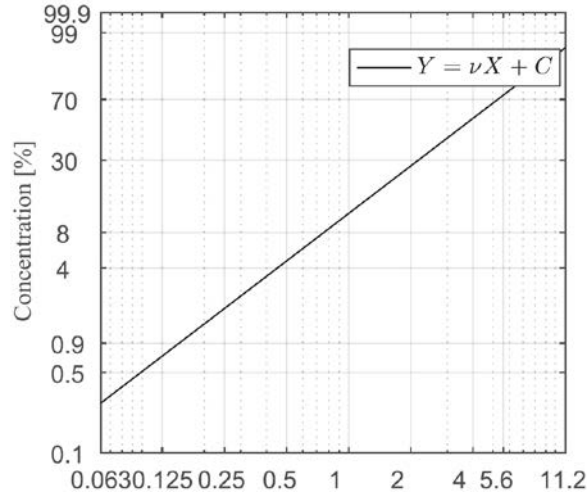


Figure 56. Unimodal Weibull function in a Weibull diagram

By examining the cumulative distribution of the concentrations of critical metals it was noted that the distributions did not form a straight line, which is why it was necessary to use a modified Weibull distribution capable of representing the bimodal frequency distribution of the element concentration. The model estimating the normalized cumulative concentration is given in equation (14).

$$\hat{H}_o(x) = 1 - \beta e^{-\left(\frac{x}{\lambda_1}\right)^{\nu_1}} - \left( (1 - \beta) e^{-\left(\frac{x}{\lambda_2}\right)^{\nu_2}} \right) \quad (14)$$

### 6.2.2 Calibration of the model

The model presented in equation (14) was calibrated against the geochemical analysis values presented in the previous section. The calibration was performed via non-linear regression analysis. The validation of the model for W2 ore is shown in Figure 57. The resulting four calibration constants for all ores are shown in Table 9. The model fits the expected outcome well. The tungsten ores exhibit less deviation in the smaller fractions. The use of log scales enhances the perception.

 Table 9. Calibration constants at  $\beta = 0.4$ 

Test	$\nu_2$	$\nu_1$	$\lambda_2$	$\lambda_1$
W1-010	0.9296	0.9960	0.1697	2.3119
W1-020	1.0681	5.4612	0.3556	4.6437
W1-030	0.9972	1.1196	0.2018	2.5603
W2-010	0.9654	1.0105	0.2037	4.4647
W2-020	1.7013	0.4056	0.1695	0.1888
W2-030	1.4658	0.5491	0.1275	2.3789
T1-010	1.2754	1.3778	0.3668	4.5945
T1-020	1.3082	2.1395	0.4341	4.7650
T1-030	1.2737	2.5134	0.4298	5.6340
T2-010	1.1573	1.5857	0.3469	4.8491
T2-020	1.2065	1.2829	0.2978	4.1769
T2-030	1.1665	1.3391	0.3362	4.9371

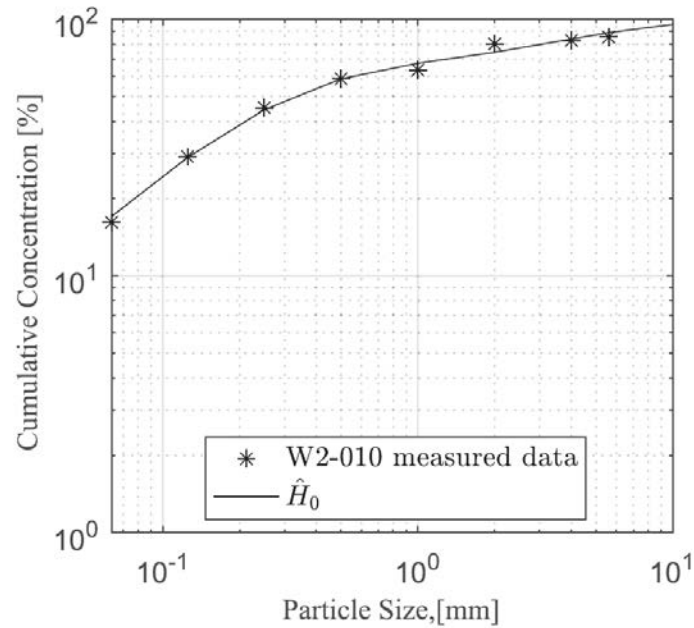


Figure 57. Calibration of cumulative concentration against W2

### 6.2.3 Application of the model

The model for simulating the absolute elemental concentration brings new opportunities and applications since increasing the resolution of coarse comminution models. Nevertheless, this model must be combined with other modelling components in the crusher model. Coarse comminution models are capable of predicting process data (output) by using mathematical models once the operating parameters (input) are known in Figure 58. Generally, the process data for coarse comminution is limited to the prediction of the energy consumption, capacity and particle size distribution. The possibility to evaluate material properties to the prediction of element concentration increase the resolution.

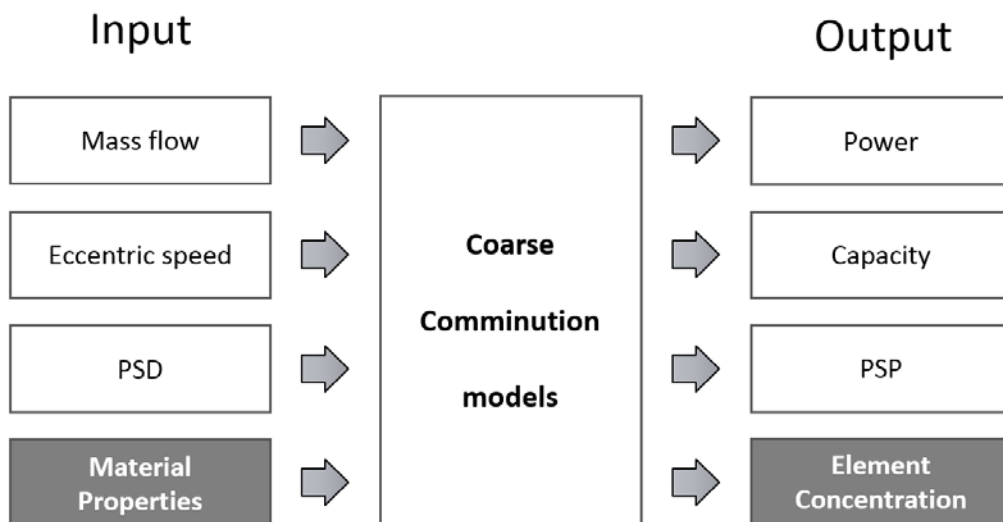


Figure 58. Predictive modelling parameters for coarse comminution.

# 7

## IMPLEMENTATION – CASE STUDY

*The aim of this chapter is to:*

- *Verify the usefulness of the developed concentration model in a case study.*
- *Describe how a plant can be optimized by using the concentration model in a cone crusher.*

The concentration model itself offers limited information, but when it is combined with other models proposed by previous researchers in Chalmers Rock Processing System, it is possible to attain more details of the process and offer improvements in the process.

The aim of this section is to demonstrate a theoretical case where the concentration model allows educated or well-based estimations of how a plant should be designed. One of the ores that were analysed was W2. The W2 plant was analysed in order to propose theoretical arrangements that could benefit the process. Figure 59 shows the configuration of the W2 plant. The process starts with a conveyor that transports the material to the jaw crushers. After the primary reduction the material goes through a second conveyor to a screen that will divide the material flow into three parts depending on the sizes. The first product will return to the primary reduction step. The second product goes to the cone crusher in order to be reduced in size again and the third product goes to the gyratory crusher. After the cone crusher the fraction between 0-10mm is fed into the ball mill, to later be classified and sent to future separation processes. The concentration model was developed for particle sizes smaller than 12 mm, and the application of this model therefore corresponds to the cone crusher.

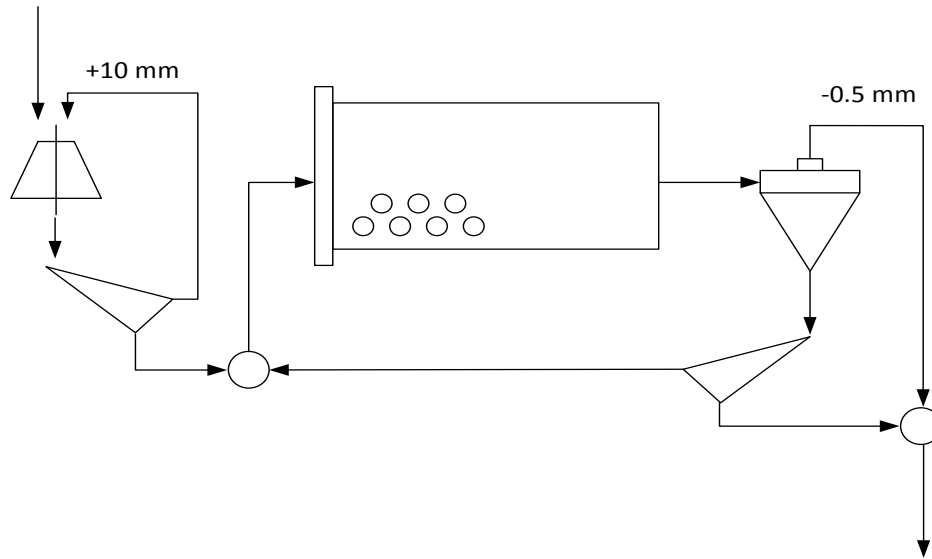


Figure 59. W2 ore dressing plant.

The tungsten distribution in different size fractions after the reduction step in the cone crusher is shown in Figure 60. The metal content in different size fractions is not homogeneous, and varies by the size of the particles. For size fractions smaller than 500 microns, high concentration of the element has been observed. This fact suggests that carrying out an early separation of the material after the cone crusher could benefit the process, but the early separation should be connected with an increase in the generation of fine particles.

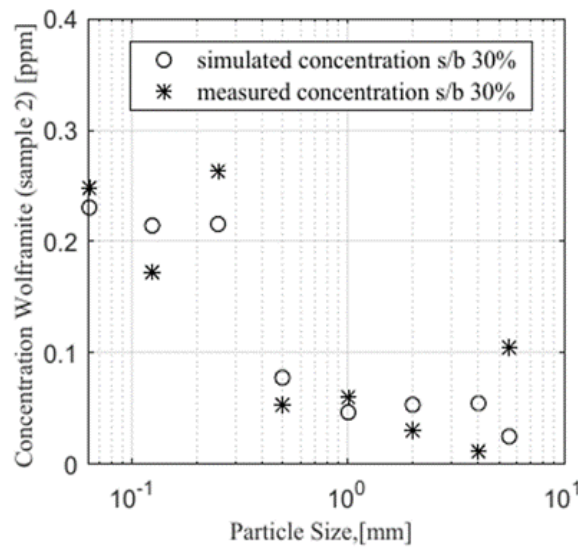


Figure 60. Predictive modelling of the tungsten concentration in W2 ore after compressive breakage.

The increase of the fine particles will be achieved by changing the machine parameters, such as the close side setting of the machine (CSS). Higher opening values will generate larger particles and closer opening values will generate finer particles. It is known that the tungsten concentration will be larger in fine particles, which is why the creation of more fine particles is desirable and hence why closer values of CSS are suggested.

The increase of fine particles must be accompanied by a screening and classification before the milling stage. Not separating this product could generate risk of overgrinding during the milling process and using more energy than necessary. The separation by size could be done by placing a classifier after the cone crusher. The classifier will allow the finest material (sizes smaller than 1mm) to be sent to separation processes such as flotation. On the other hand, the largest particles (sizes larger than 1mm) will be sent to the milling process in order to be reduced in size. The fraction between 0 and 1mm represents 18% of the total fraction. By reducing the mass flow by up to 18 % into the mill a huge amount of energy could be saved.

Evidently the integration of a new machine will generate an investment cost in equipment and additional maintenance, but this could be solved by rearranging the equipment that the plant already has. Figure 61 and Figure 62 show both options of rearrangement, case one by adding an extra classifier, and case two by moving an existing classifier.

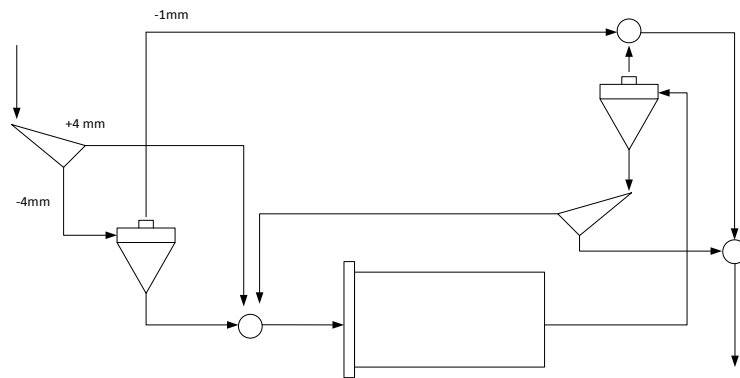


Figure 61. Theoretical suggestion for rearrangement of W2 plant. Case 1, additional pre-mill screening.

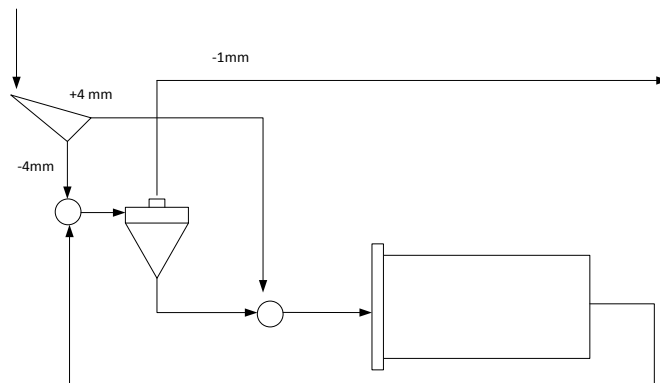


Figure 62. Theoretical suggestion for rearrangement of W2 plant. Case 2, pre-mill classifier.



# 8

## DISCUSSIONS AND CONCLUSION

*The aim of this chapter is to:*

- *Present the most important conclusions drawn in this thesis.*
- *Answer the research questions stated in Chapter 2.*
- *Discuss what has been found as important for future work but not researched.*

The purpose of this work was to investigate how ore characteristics can be measured and integrated into coarse comminution models, followed by developing a test procedure capable of predicting the critical metal concentration as a function of the particle size distribution after a compression cycle in a crushing process.

The main hypothesis of this research stated that critical metals are not evenly distributed in different size fractions during coarse comminution processes, since the breakage of particles is affected by the mineralogy and texture. Furthermore, it has been demonstrated how significant the impact of having a better understanding of mineralogy has when working with breakage.

### 8.1 GENERAL

The research results that have been discussed aimed at increasing the platform for modelling the compressive breakage. A test procedure capable of analysing ore characteristics after compressive breakage forces has been integrated into coarse comminution routines. The procedure includes mechanical characterisation of the particles after compressive breakage as well as mineralogical analysis through the use of a Scanning Electron Microscope and element concentration completed by geochemical analysis. The procedure is capable of measuring the element concentration and identifying minerals in different size fractions after a cycle of the compressive crushing sequence. Moreover a model with the capability to analyse the critical metal concentration as a function of the particle size distribution has been developed. In order to achieve this, four ores of tungsten and tantalum were evaluated and characterized.

Some general conclusions are formulated below:

- It is possible to achieve liberation of critical metals after coarse comminution processes, however the amount of liberated particles is low.
- The developed test methodology, allow the interpretation, evaluation and comparison of different ores with different behaviour and properties by acquiring similar parameters.
- Different types of ores with different mechanical and chemical behaviour show similar properties that can be analysed and modelled for, for example, the critical metals content.
- Different types of ores with different mineral composition show the same type of breakage and accumulation of minerals in different fractions, such as for example, an accumulation of micas and critical metals in fine particle size fractions.
- The distribution of critical metals after a series of compressive breakage processes has been evaluated. It has been observed that the critical elements are not evenly distributed in different size fractions during coarse comminution processes.
- It is possible to model the critical metal content in different size fraction after a series of compressive breakage processes.
- Plant performance and cone crusher performance could be improved by incorporating the composition model.
- The resolution of the comminution models has been increased by the addition of the concentration model. The material properties could be used as an input parameter, to get the element concentration as an output.

## 8.2 ANSWERS TO RESEARCH QUESTIONS

The following answers are given to the research questions stated in Chapter 2 of this thesis:

*RQ 1)* What type of material properties beside particle size need to be analysed in order to model element grade?

It is necessary to analyse more properties in addition to particle size distribution. An ore is an aggregate of minerals. When an ore is broken and divided into different particles, each of these particles is going to have different composition. Each particle will be constituted by different minerals in different portions, therefore the element grade will vary in each of them. By analysing element concentration in different particle size fraction it is possible to construct a model that reflects what occurs in each size fraction.

*RQ 2)* How can liberation in coarse comminution be measured?

The liberation during coarse comminution process can be investigated through the use of a Scanning Electron Microscope. SEM is a qualitative method in which it is possible to examine and identify mineral composition in the particles. During the analysis it is possible to evaluate how a particle is composed, if it is integrated by one mineral or if it is a composite particle. When a particle is integrated by only one mineral it is considered to be liberated, and this is possible to analyse by the use of SEM.



*RQ 3)* Is it possible to achieve liberation of critical metals in coarse comminution?

Yes, it is possible to achieve liberation of critical metals minerals during coarse comminution processes. It was demonstrated that after a repeated compression at different compression ratios, it is possible to find liberated particles in sizes fractions finer than 0.125 mm.

*RQ 4)* What factors influence the concentration distribution and how can it be measured?

The element concentration could be measured by performing a geochemical analysis of the rock. The geochemical analysis is a qualitative method that measures the content of different elements in the rock sample. By analysing the element concentration in different size fraction particles, after compressive breakage, it is possible to analyse the distribution of the critical elements.

When an ore is broken and divided into different particles, each of these particles is going to have different composition. Each particle will be constituted by different minerals in different portions, therefore the element grade will vary in each of them. By analysing the element concentration in different particles size fractions, after compressive breakage, it is possible to analyse the distribution of the critical elements.

*RQ 5)* How can concentration of critical metals be modelled?

A model procedure has been developed where it is possible to predict the concentration of critical metals after a series of compressive crushing. The critical element concentration needs to be normalized and presented as cumulative distribution. The normalized cumulative element concentration can be modelled by using a bimodal Weibull distribution.

*RQ 6)* How can coarse comminution improve the extraction of critical metals?

An industrial case study has demonstrated where in the process improvements can be made to enhance the performance. By using the concentration model in a cone crusher from the plant, it was perceived that in finer particle sizes the concentration of tungsten was higher. To ensure an improvement it is first necessary to change machine settings in the cone crusher in order to produce finer particles. By then adding an extra screening and classifying particles by size before the milling stage the finest material (sizes smaller than 1mm) will be sent to a separation processes such as for example, flotation. The largest particles (sizes larger than 1mm) will be sent to the milling process in order to be reduced in size. By reducing the mass flow into the mill, energy could be saved.



# 9

## FUTURE WORK

*The aim of this chapter is to:*

- *Discuss what has been found important for future work but so far not researched.*

The work presented in this thesis has been dedicated to the development of a test procedure capable of measuring ore characteristics and integrating them into coarse comminution models. A model was developed which has the capability to predict critical element concentration after repeated compression at different compression ratios. This model could be expanded by analysing the behaviour of major elements and proposing a model capable of predicting their concentration as a function of the size particle.

Moreover, element concentration is not the only ore characteristic that could be measured and integrated in coarse comminution models. It was observed that minerals are also not evenly distributed in different size fractions after compression breakage, but this affirmation was not quantified. Quantification of minerals in different size fractions is necessary to develop new models that relate mineralogy with particle size distribution.

Another characteristic that needs to be quantified is liberation of particles. It was observed that it is possible to achieve liberation during coarse comminution, however it is necessary to have quantitative data in order to develop a liberation model for coarse comminution. Quantitative data of liberation and minerals can be obtained by performing mineral liberation analysis (MLA) or Quantitative Evaluation of Minerals by SCANNing electron microscopy (QEMSCAN).

The concentration model was validated for low grade ores of tantalum and tungsten. The next step could include the validation of this model for other types of ores, as well validating the possibility to apply this model to high grade ores.

A plant validation is needed for future work. Working with real data will increase the value of the concentration model and its possibilities. By analysing real cases and real data it will be possible to validate the usage of the model and its capacities.

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